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LIST OF ACRONYMS

CTE – Coefficient of thermal expansion
CPM- Crucible (Research) Powder Manufactured
FS – Flame Sprayed
HDI – High Density Infrared
HVOF- High Velocity Oxy-Fuel
IR - Infrared
TLC-Transient Liquid Coating

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1.0 EXECUTIVE SUMMARY

A 4 year, \$350,000 / yr effort to develop high density infrared (HDI) coating methodology that could replace furnace or flame fusing was the subject of this investigation. The effort was a collaborative effort between Oak Ridge National Laboratory (ORNL), Materials Resources International and an industry team of participants. The project's aim was to develop, evaluate and understand how high density infrared heating technology could be used to improve infiltrated carbide wear coatings and/or to densify sprayed coatings. In the proposed work both applied and fundamental investigations were conducted. The applied work at ORNL aimed at developing practical High Density Infrared (HDI) heating systems and techniques that were able to fuse thermally sprayed coating and to infiltrate carbide coatings for use in applications such as agricultural blades. The expectation was that the developments would also have application in coating rolls for metallurgical processing, in coating of components used construction and mining vehicles, components for paper and polymer processing. Engineering development focused on developing process technology and know-how to implement HDI systems that could fuse coatings on a range and size of components. Fundamental research and process modeling was conducted that developed some understanding the interaction of HDI processing with the coating materials and the base materials, and determined selected coating properties, focusing in particular on wear resistance.

The research included HDI fusion evaluations of infiltrated carbide suspensions such (BrazoCoat® S), composite suspensions with tool steel powders, thermally sprayed Ni-Cr- B-Si (self fluxing alloy) and nickel powder layers. One basis of the research was that there might be significant growth in fused cermet coating technology on larger components, if only the surface of parts had to be heated through intense local surface heating. A major project aim was to compare this new class of thin, HDI fused cermet/carbide to coatings fused by more conventional methods.

The project has developed an understanding of how high density infrared heating technology can be applied and how it can be used to improve infiltrated carbide wear coatings and thermally sprayed coatings. The project has had both applied and fundamental components to the investigations. The applied work has developed practical HDI / transient liquid coating (TLC) procedures on test plates that demonstrate the ability to fuse carbide coatings for industrial applications such as agricultural blades, construction and mining vehicles. Fundamental studies have advanced process models that have led to improved process understanding and control.

Process Development

The HDI process understanding was advanced and the behavior of various materials including nickel, tool steel and reinforced wear coatings consisting of NiCrP and carbides were studied. ORNL conducted process modeling and developed a model that enabled the study of IR beam interaction with surfaces and molten layers, resulting in several processing changes that permitted the HDI process development to be shortened. The modeling efforts, later in the second year determined the importance of lower power beam preheat before the high power beam irradiated the surface melting. The preheat passes were found to bring the average temperature of the surrounding base metal to a regime where when the center beam area went molten, the molten metals wetted and adhered better and better coating penetration was achieved. Process studies evaluated beam power, traverse speeds and atmosphere controls; all aimed at achieving the densest possible coatings from a range of coating materials and precursor layers types, such as thermal spray and suspensions. The process development showed that stainless steels, tool steels and nickel alloys, without the addition of wetting agents such as Boron, Phosphorous, and/or Silicon, did not adhere as well to the underlying base metals. These results showed that coating local chemistry which can affect

local surface wetting played an important role in HDI's rapid fusing capacity. The project concluded with a much better understanding of the HDI process; however, process control of final coating properties were not optimized nor did experiments prove the robustness of the process for composite suspension claddings. The interaction of materials to the HDI beam and process variations were more complex than was able to be modeled.

Success & Limitations

Very good abrasive and sliding "carbide-tool steel" composite suspension wear coating properties were produced; however the repeatability of the process on making carbide reinforced coatings, in the end, was not demonstrated. Additional work is recommended based on the excellent, but variable wear results. Due to time and funding constraints, evaluations of HDI densification of thermally sprayed corrosion and wear coatings, although promising, were not able to be completed. The HDI process did show promise in its ability to densify thermally sprayed WC reinforced coatings and stainless steel. HDI was shown to be a very broad, intense heating process that can surface melt layers, but the properties of the pre-HDI processed layers had a large influence on the HDI processing parameters. Suspension coatings had a binder and low thermal conductivity while thermally sprayed layers had much higher densities but normally were much thicker. The HDI power and traverse rate needed to be adjusted and variations in the underlying structures led to coating densification differences. One limitation is that the HDI process robustness is dependent on the precursor materials and their structures. It was also found that wetting of underlying layers was affected locally by chemistry and that surface molten metal wetting elements such as B, Si and P were beneficial in achieving adherent HDI processed coatings. If metal layers were already intimately bonded, such as in High Velocity Oxy-Fuel (HVOF) coatings, then coating adherence was not found to be as factor. The HDI process was indeed capable of fusion and in the case of thin carbide reinforced suspension applied coatings, excellent coatings could be achieved. However; due to the binder and poor thermal conductivity of the suspensions precursors, coating thicknesses much over 0.2 mm (0.005") were not able to be densified.

Wear Results

Suspension applied carbide composite coatings, 0.002" – 0.005" in thickness and thermally sprayed "spray and fuse" NiCrBSiC coating were able to be effectively HDI processed. Time and funding constraints limited detailed wear studies to these types of coatings. Both abrasion and sliding wear properties were measured and comparisons to other coatings showed that excellent "specific" wear rates were able to be achieved. In particular, the thin composite claddings that combined WC carbide, NiCrP binders and tool steel particles proved to be the best wear coatings, creating almost negligible wear rates in an ASTM G 77, Ranking Resistance of Materials to Sliding Wear run a Falex compared to bulk tool steel and even to HDI processed flame sprayed Ni-Self Fluxing (NiCrBSiC) coatings. Under Pin Abrasion Test (ASTM G-132) and Sand Abrasion (ASTM G-65), the same HDI processed composite tool steel / carbide reinforced coating performed very well. The project has demonstrated the relatively thin composite wear coatings with additions of tool steel (Crucible Rex 121) are excellent candidates for both sliding and abrasion wear resistance. HDI fuse flame sprayed Ni-Self Fluxing (NiCrBSiC) coatings were also superior to furnace fused coatings.

Conclusions

The results of the process investigation concluded that:

- HDI can fuse and densify a range of coating materials and types
- HDI process will be sensitive to the underlying precursor compositions, structures and thicknesses.

- HDI process modeling was successful in improving process parameters but the model's ability to predict process interaction with coating properties was not achieved.
- HDI process control for coating densification, although improved, was not optimized. Too many variables in pre-layer structures interacted with the ability to adequately control final final coating structures. Repeatability was not established to the degree needed for an industrial process.
- The project did show that HDI has the potential to be an effective replacement for furnace heating, thus accomplishing a major objective of energy conservation, provided the HDI process can be scaled to more complex geometries.
- This particular project was not able to study the scaling of the HDI process from smaller, flat ~ 3" x 3" coupons up to larger plates and cylinders.
- The HDI process was able to produce excellent wear coatings, especially when processing carbide reinforced-tool steel coatings from suspensions.
- It is recommended that additional work to scale the HDI process to larger plates and cylinders be conducted, concentrating on the carbide-tool steel composite coatings. Once scaled to larger components, then the HDI process will have achieved its potential for enabling larger structures to be effectively coated with highly wear resistant layers.
- It is also recommended that the HDI process variability be better understood; focusing on the carbide-tool steel composite suspension precursors systems. Based on wear testing, these materials showed the most promise as an industrial coating, provided that the HDI process be scaled to heat and fuse coatings on larger plates and cylindrical shapes.

2.0 BACKGROUND

2.1 High Density Infrared Processing vs. Conventional Processing

ORNL has been conducting research and development on plasma lamp materials processing with Vortek Industries. This arc lamp is depicted in Figure 1a) and thermal processing that utilizes the radiant output of this lamp has been termed, high density infrared processing. ORNL has developed a development laboratory and user facility with this equipment. Figures 1b) and 1c) show these ORNL facilities. One can see the HDI lamp mounted on a large five-axis robotic manipulator arm. Test sample processing is done below the lamp and in controlled atmospheres using a box with a quartz window cover to permit processing of materials in a controlled atmosphere. An x-y table and a lathe bed to move parts while heat-treating or fusing coatings is installed in the ORNL processing facility. Another feature of the plasma lamp for this facility is a water window in directly in front of the lamp. This water window device passes a thin film of water over the quartz glass covering the elliptical reflector, as seen in Figure 1b . This feature protects the lamp when operating in harsh environments. This widow has a 3-mm water film that continuously cools the lamp quartz window. This water window also protects the lamp from liquid metal splatter, which is necessary in many processing applications.

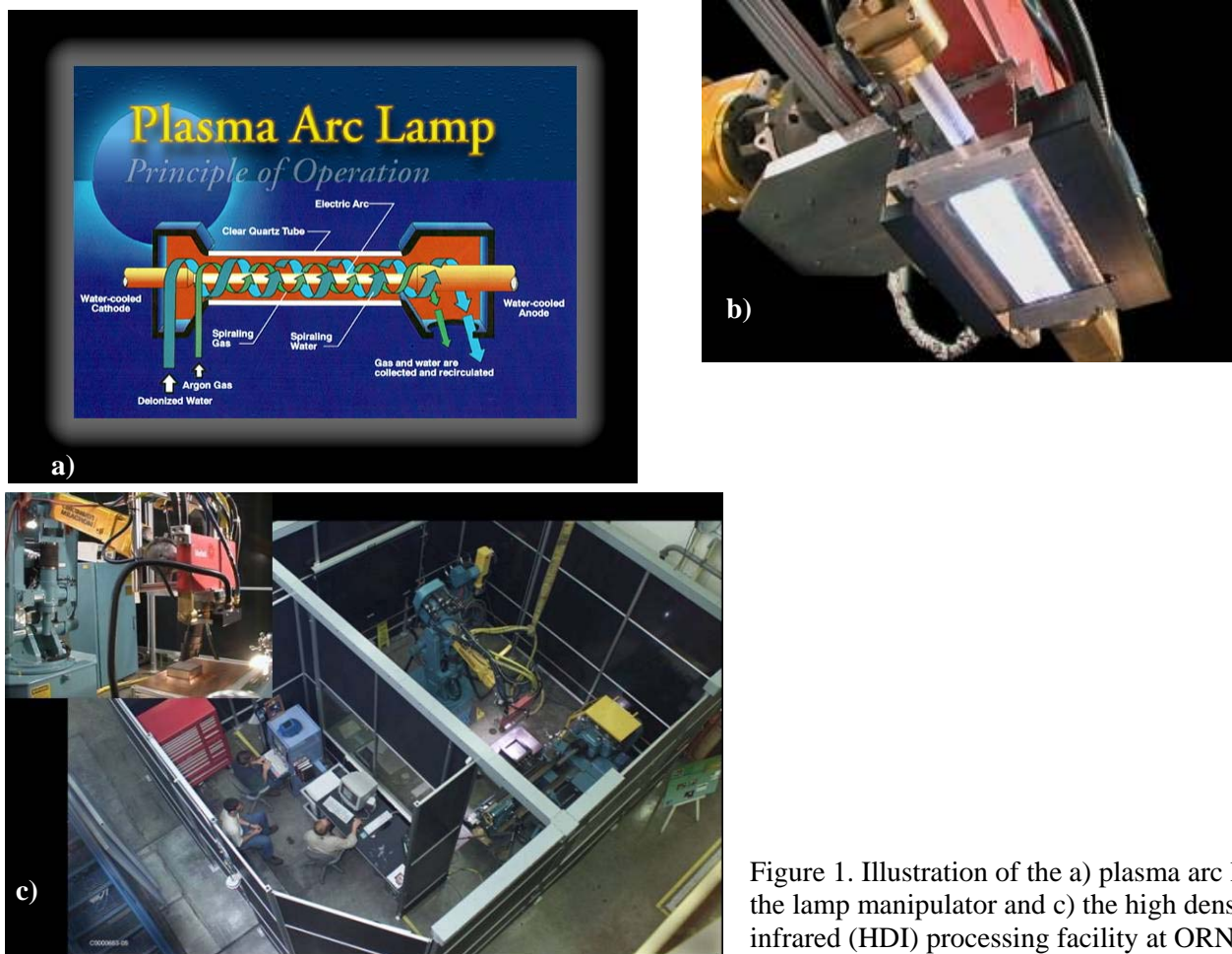


Figure 1. Illustration of the a) plasma arc lamp b) the lamp manipulator and c) the high density infrared (HDI) processing facility at ORNL.

HDI is an intense surface heating process similar to laser heating. However, the extremely large power densities used in laser fusing forces convective stirring in the molten pool adding to higher local heating that decomposes many second phase hard particles. Additionally, in laser processing, the small beam size creates many more overlap areas which may make the process impractical for large component work. Currently, industrial thermal sprayed coatings are typically fused with either flame or furnace fusing techniques. HDI promises to provide more intense surface heating, enough to locally fuse coatings without the need to place large components into furnaces thus increasing the application of fused coatings, while providing significant energy savings.

In this work, coating precursor materials were applied via suspension spraying or were thermally sprayed. These precursor layers provided the materials for fusing and were carbide/cermet or NiCr-B-Si-C materials. Such coatings have been historically applied to surfaces for wear and corrosion protection via weld surfacing or thermal spray. The disadvantage of weld surfacing is it can only be accomplished on metals that are not affected negatively by the welding heat effects and/or the dilution of the base materials by the weld. These weld overlay coatings also have volume percent carbide limited to approximately 20v/o. Thermal spray coatings increase wear resistance with their increased carbide particle contents, up to 50 volume percent (v/o). These increases in carbide content become limited in many high wear applications by the fact that thermal spray coatings are not metallurgically fused. Under high wear pressures, typical in rolls and dies, thermal spray coatings spall unless fused. In these applications fusion is required to better bind the carbides in the coating or bond the coating to the base materials. Such thermally sprayed coatings fused in furnaces or by flames have been shown to have lower carbide content than as deposited thermal spray (e.g. HVOF or plasma) coatings. One should note that large fusion layers and times lead to the segregation of carbide particles (WC, TiC, Cr_xC_y , etc.). The HDI coating process developments in this project were aimed to effectively fuse the thermal spray and/or suspension applied carbide reinforced coatings in order to retain a high percentage and uniformly distributed carbide content that would increase the wear resistance of coatings.

MRi and several of the industrial partners have developed cermet material precursors. MRi has worked with its partners to introduce a carbide / braze filler metal suspension system that brings a high volume percent carbide cermet mix to a surface that requires fusion in a protective atmosphere to keep the boron, silicon and the chromium elements from oxidizing. The proposed HDI / TLC process has shown itself to have the potential to complete this coating material system in a protective shroud gas environment while enabling larger parts to be effectively and economically coated. In preliminary testing, the suspension / cermet coatings that MRi has been developing, was shown to enhance the efficiency of HDI fusion due to the black mat / black body surface that the suspension creates to more effectively absorb the HDI IR radiation.

The 4 year R&D effort, being reported here, aimed to show that suspension cermet and higher content cermet thermal spray and fuse coatings introduced over 15 years ago have the potential to be applied to larger components that cannot be subjected to high surface or bulk temperatures ~ 2100°F, normally used to fuse and/or infiltrated the coatings. The economics and energy efficiencies of heating the bulk of the larger components are not attractive and it was proposed that the HDI processes could be used to surface fuse coatings on large components. A short duration, localized heating method such as HDI would also be compatible with using localized protective atmospheres and thus see wide spread application for advanced cermet coating materials systems.

The project studies were aimed at understanding equipment control, process control, and metallurgical control issues. For example, understanding the power and scanning requirements for HDI fusing of loose powder base materials on a substrate surface and the influence of convective stirring which limits heat transfer to the underlying substrate. The understanding of the process interaction fundamentals (liquid formation, convective stirring, infiltration, wetting, heat transfer, and solidification) in coating precursors and their interaction with HDI radiation were evaluated using modeling and process experiments.

The project's aim was to develop a new surface heating tool that would be able to heat a large surface area simultaneously to permit the fusion and/or braze infiltration of carbide coating precursors. The primary coating precursors to be evaluated were cermet suspensions and thermally sprayed coatings which require further densification and base metal fusion. Improved HDI technology would be a new tool to apply the commercially available wear coatings to replace certain chromium hard coat coatings, non-fused flame spray coatings, and welded hard face surfaces. These type coatings also offer an opportunity to be much more wear resistant than many commercially available coatings due to the high, well dispersed, very fine carbide distributions. HDI infiltrated coatings have the potential to be very thin and well controlled, thus conserving both energy and expensive materials as well as eliminating chromium plating processes that contain high levels of hexavalent chromium.

The industries where HDI wear coatings would offer benefits and savings are highlighted below.

Mining:	earthmoving, material transfer systems
Agriculture:	blades – (cutting corn, harvesting, etc.)' bio-mass gasification systems
Glass:	transfer lines (wear resistant); cyclones; (corrosion; wear)
Petroleum:	pump body housings , pumping slurries (corrosion)
Steel:	boilers, weld overlays , steel processing and transfer rolls steel – hot rolling cold rolling
Metal-casting:	corrosion-resistant coatings for die casting (extend die life 100%)
Forest Products:	wood yard (grinding) – harvesters (pinchers); slurry pumps
Energy:	boilers , corrosion-resistant coatings as fireside of gasifiers boiler tubes and bio-mass gasification systems

3.0 TEST PLAN AND METHODS

3.1 Introduction

The HDI Equipment

The HDI / TLC process utilizes a unique technology to produce extremely high-power densities of 3.5 kW/cm² with a single lamp, which is currently one of the most powerful arc lamps in the world. Instead of using an electrically heated resistive element to produce radiant energy, a controlled and contained plasma is utilized. The lamp consists of a 1 - 2.5 cm diameter quartz tube, which can be 11.5, 20, or 35 cm long. The lamp is sealed at the ends where the cathode and anode are located. De-ionized water mixed with argon or nitrogen gas enters at the cathode side through high-velocity jets impinging at a given angle. Due to the high velocities and pressure, the de-ionized water is impelled to the wall of the quartz tube and spirals down the length of the tube in a uniform 2- to 3-mm-thick film. This water film serves two purposes: (1) to cool the quartz wall and (2) to remove any tungsten particulate that may be expelled from the electrodes. The gas moves in a spiral fashion through the center of the tube, and a capacitive circuit initiates the plasma. The plasma, which has a temperature in excess of 10,000 K, is stable and produces a radiant spectrum from 0.2 to 1.4 micron. The spectrum is primarily in the infrared (0.78 to 1.00 μ m), although substantial energy is released in the visible wavelength, similar to the appearance of natural sunlight in energy distribution and color rendition.

The output high intensity IR spectrum is absorbed with high efficiency by metal surfaces. In contrast, the spectrum of a CO₂ laser with wavelengths near 10.6 micron is absorbed with much lower efficiency. Powder coatings discussed here are highly absorbing because the open areas act like black bodies. The lamp has a typical life of in excess of 500 h, and failure occurs in the anodes and cathode, which are inexpensive and can be changed in approximately 15 min. Furthermore, the lamp has a consistent spectral output independent of lamp life and power level and convert electrical energy into radiant energy in excess of 50% efficient. The lamp is typically configured with a reflector to produce a line focus or an area of uniform irradiance. The characteristics of the HDI process of particular interest in large area coating fusions consist of:

- Large area coverage, 3.175 cm by 10 to 35 cm in a line focus or up to 10 cm by 20 cm in an uniform irradiance.
- Limited convective mixing of the coating material with the base material.
- Rapid cooling of the coating materials.
- Minimal effects on the base material.
- Carbide reinforcements in the coating are not degraded.
- Virtually no temperature limitation, can readily melt tungsten.
- HDI provides a rapid, localized heating method that can enable the use of advanced fused cermet coatings on large industrial processing systems.
- Surfaces are heated sufficiently to fuse and metallurgically bond coatings to components

3.2 Technical Objectives

The project goal was to develop, evaluate and understand how high density infrared heating technology can be used to improve infiltrated carbide wear and thermal sprayed coatings. In the proposed work, both

applied and fundamental investigations were conducted. The applied work focused on developing practical HDI processes that would be able to fuse carbide coatings for industrial applications such as agricultural blades, rolls for metallurgical processing, construction and mining vehicles, components for paper and polymer processing as well as for a range of cross cutting applications. Engineering development thus centered on developing the process and equipment technology necessary to implement industrial HDI systems that can fuse coatings on such parts. Fundamental research was aimed at understanding the interaction of HDI processing on the candidate coating materials and their subsequent properties.

The scope of the work included the following:

Coating – Base Metal Interactions: Understand the basic coating-base metal property changes and the influence of coating precursor impurities, thermal conductivity, emissivity, on the coating structure and bonding as a function of the precursor deposition techniques, powder suspensions, powder mats, and thermal spray. This work was a combination of modeling and experiments designed to understand the basic elements of the HDI/TLC processes.

HDI Process – Materials Interactions: Develop understanding of the interaction of the radiant beam with the precursor; evaluating i) melting, ii) melt pool movement (laminar and convective), iii) liquid-solid interactions (particles and substrate) and iv) fused layer solidification.

HDI Interaction and Heat affects on Base materials: Understanding the effects of the fusing cycles on the base materials.

Coating Test and Evaluation: Develop coating properties data including surface roughness, bond strength, and wear resistance.

Industrial Coating Demonstrations: Work with the industrial partners to identify high potential applications and coat components using the developed HDI processes and assess the most suitable precursor for that application. These components will be coated and evaluated in service with the appropriate industrial partner.

Process Control and Optimization: After understanding the basic processes, models and experimental results were incorporated into a process control logic. Process and coating precursor (suspension, mat or thermal spray) parameters were to be optimized for the most improved coating properties.

These objectives were to be achieved through the following tasks:

1. **Industry Survey:** This task was to be executed early with the participating industrial partners and be used to determine the scope of the coating requirements and the range of applications to be developed and studied over the three year project.
2. **Materials/Precursor Development:** This task was an iterative task between Tasks 3.0 and 4.0. In this work; processing, structure and property evaluations were to be made on coated coupons from which metallographic, mechanical, wear and corrosion test specimens were to be made in order to:
 - a. Identify the appropriate material or materials systems to give the resultant wear and corrosion properties for ferrous base materials in the identified application. These

- coatings include highly abrasive and erosive wear that rely on WC, Cr_3C_2 , , mixes and/or oxide particles that will be combined with NiCrSi(B) and NiCr(P) metallic matrices.
- b. Investigate different precursor deposition techniques. These included suspensions, mats and thermal sprayed layers.
3. **HDI Process Studies:** This task aimed to develop the HDI process for fusing the coatings. The work was to:
- 3.1 Identify the mechanisms and HDI processing parameters, power and scan speeds, to produce coatings with enhanced wear and corrosion properties for industrial applications.
 - 3.2 Evaluate these coated coupons as part of Tasks 2.0 and 4.0.
 - 3.3 Develop a process model that links the HDI process parameters to coating structures.
4. **Coating Test and Evaluation:** This task determined the coatings structures and processes via:
- 4.1 Metallurgical Analysis: section, polish and document the coating structures on coupons coated under Task 2.0 investigations. Both optical and SEM analysis were to be used to assess coating interactions with the base metals, the range of coating phases and the stability and distribution of the wear resistant carbide particles.
 - 4.2 Conduct hardness testing and
 - 4.3 Conduct abrasive and sliding wear tests
5. **Field Testing:** This task is aimed to demonstrated the potential that the developed HDI coatings using the following industrial partners and testing.
- 5.1 Lund International: is engaged in the development and commercial production and sale of agricultural blades. This task was to coat an assortment of smaller foraging harvester blades used in large agricultural combines and any other blade that may see improvements stemming from HDI processes.
 - 5.2 Ametek /Crucible / Carpenter: are powder producers that will be used in the precursor layers. The project will work with these companies to investigate coatings that utilize some of their latest materials and work with their customer/end users

3.3 Approach

The project was divided into two major efforts, Year 1) Scoping studies of materials and process and Years 2 -4) Basic Process Studies and Materials Evaluations.

3.3.1 Initial Approach / Scoping Studies / Year 1

In the initial Year 1 study the various industrial partners offered their materials and applications for initial evaluations. These initial tests concluded:

-
- Initial HDI experiments produced metal and cermet coatings on steels but with varying degrees of porosity and adhesion but the tests were encouraging
- These initial tests showed that for Ni-Self Fluxing coatings that hardness improvements were achieved.
- Surface hardening of Tool Steel Alloy (CPM 10V) provided by Crucible Research. The initial infrared surface hardening experiments indicated that surface hardening could be achieved in tool steels without overlay coatings, utilizing the high quench rates of surface heated layers.

- Tests showed that wetting of base materials by the IR melted layers was variable depending on the composition of the precursor layers being melted and fused.
- HDI trials on a range of thermal spray coatings (flame, plasma and HVOF) showed the sensitivity to structure of precursor layers due to precursor layers' porosity, oxide content and layer bonding. All of these factors were found to lower the "fusing" layers' local thermal conductivity.
- The issues identified in Year 1 were:
 - 1.) Inconsistent melting of the thermally sprayed precursor layers;
 - 2.) Lack of wetting of the steel substrates by the molten layers; and
 - 3.) Inconsistent HDI fusion and densification of coatings.

These issues led to a focus on fundamentals and effort to develop an HDI / Coating-Substrate Process Model. The modeling effort required measurements of precursor layer thermal properties (specific heats [C_p], and thermal conductivity [k]) and an evaluation of thermal spray coating structure with their respective thermal properties. Understanding the coating properties would permit computation of coating thermal profiles upon which a working model of the HDI coating process could be developed. After the computer modeling was completed, coatings were to be made and optimized based on depth of fusion, density, coating bonding and coating structure.

3.3.2 Approach / Years 2-4

Focus on "Model" Material Systems (precursor / substrate)

These alloys were selected as base coatings based on industrial partner input

- Ni-CrBSi Flame Spray Coating
- Stainless Steel HVOF (high velocity oxy-fuel) spray coating.
- Tool Steel (CPM) by HVOF
- Suspensions with Carbide and/or Tool Steel reinforcements

Characterize Coating

The coating precursor materials layer properties and characteristics were needed to develop initial starting properties of the layers prior to IR heating. The purpose of this set of tasks were to:

- To evaluate bond integrity of coating-substrate interface
- Evaluate/Determine thermal diffusivity
- Characterize coating-substrate interface wetting behavior/ characteristics as a function of coating-substrate combination(s)

Thermal Spray layers

The thermal spray process had its own variables and the coatings as precursor layers needed to be understood. To do this and to set up the initial conditions for HDI fusion, the thermal coatings were evaluated for:

- Density / bonding
- Characterization of Post HDI Layers
- Bonding / Structure

Thermal Modeling

A thermal computational model that related to the specifics of the HDI fusion process included the following steps:

- Prediction of IR processing parameters based on experimental data from Thermal Diffusivity measurements
- Determination of the temperature (T) where coating enters liquidus phase field
- Evaluation of the effect of CTE Mismatch (E.g., between steel and stainless steel)

- Conduction of heat transfer experiments
- Conduction of controlled IR experiments in small IR furnace with thermocouples to determine heat transfer for selected C-S systems
- Measurement of precursor layer thermal properties (C_p , k , α , and H_f)
- Evaluation of thermal spray coating structure with thermal properties
- Developing thermal profiles based on the model and layer properties.

Wear protection and commercial applications,

The success of HDI and its commercial use would depend on the performance of the various HDI processed coatings. The wear properties of these coated layers became the focus of the investigation centering their use in on:

- Agricultural Cutting Blades (for Lund International) and
- Wear Coating layer for use on rolls and glass production / tools

4.0 RESULTS & DISCUSSIONS

4.1 Task 1.0 Industry Survey

A comprehensive survey of the industrial application for HDI fused coating was not conducted. It became clear that to the industrial partners that surface wear resistance was their highest priority for development. The industrial application that received the most attention was agricultural blades with input and the participation of Lund International. MRI's interest was in application of its carbide suspension products which has application in metallurgical, polymer and glass manufacturing industries

Wear coatings for these industries are typically are overlay coatings that are intrinsically hard or have hard phases, such as carbides in a tougher metallic matrix. These coating, especially when thin, typically need to be fused to bind the hard phases and to bond to the underlying base metals such as steel. The industrial partners early in Year 1 determined that in industry survey would not provide specific benefits and that the focus of the investigation should be in process understanding and developing coating properties.

4.2 Task 2.0 Materials/Precursor Development:

Two different types of precursor layers that would be HDI fused were selected. These precursor layers, once placed on the surface, were to be rapidly melted using IR radiant heating to the point of fusion. The very high-power densities achievable with the arc lamp permit the surface melting of almost any material. The two coating methods, suspension (spray) and fuse and thermal spray and fuse, are described below. The almost instantaneous on/off capability of the high-power density arc lamp system allowed for good surface thermal profile control. In the completed work, the solid/liquid phase reactions that occurred on HDI fused surfaces were modeled and their interaction effects on the candidate base materials were studied. Initial Year 1 investigations results are presented below.

Powder Spray and Fuse

Coupling HDI processing with powder "suspension" spraying at ambient temperature is a viable method as shown in Figure 2. The figure clearly shows that suspension (sprayed) cermet ($\text{NiCrP} + \text{WC}$ or Cr_2C_3) coating could be sprayed and HDI fused without the degradation of the tungsten carbide particles.



Figure 2. High-density-infrared transient-liquid-coated steel, 15-μm tungsten carbide/Ni-P coating from a suspension precursor.

The process had not been studied at length in Year 1 and a basic understanding of the fusing process, including the influence of convective stirring, was found to be needed in order to achieve process control and reduce experimentation. Room temperature spray processes were used to deposit a suspension of WC and Cr_2C_3 with an alloy matrix on the surfaces of wear and corrosion-sensitive parts. The ceramic particulate-braze alloy layers had volume fractions as high as 50 vol. % while the matrix provided sufficient coefficient of thermal expansion to accommodate the differences in expansion and contraction between the coating and matrix.

Plasma Spray or Flame Spray and Fuse

Plasma spraying is a coating process in which powder particles of the coating material are melted in gas plasma and propelled onto the substrate surface. Coating porosity and the interfacial properties of plasma-sprayed coatings are areas that have limited the application of these coatings due to the ability of corrosive environments to penetrate the coating. In order to correct such deficiencies, it has been found that plasma-sprayed coatings can be re-melted and interface properties improved by utilizing the HDI process. A plasma-sprayed coating that has been HDI processed is shown in Figure 3.

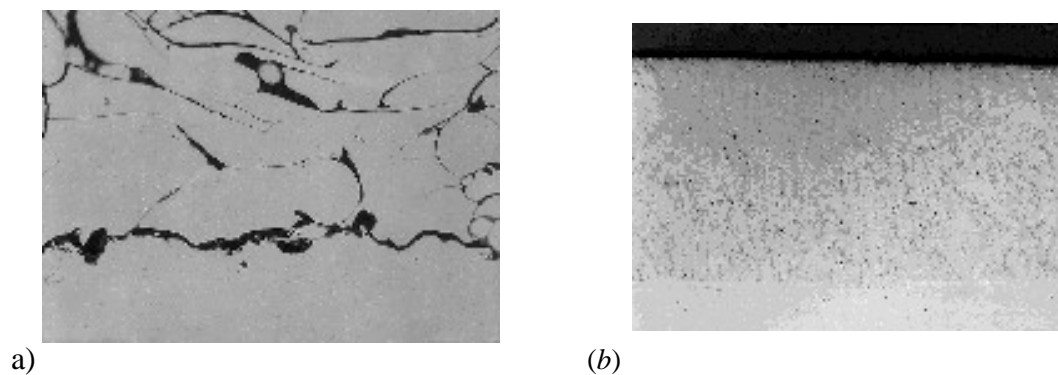


Figure 3. (a) Hardfacing alloy as-thermal sprayed and (b) after high-density infrared-fused at 1000 W/cm^2 0.5 cm/s.

The HDI processed thermal sprayed coating has dramatically reduced in coating porosity as seen in Figure 3. The coating only has micro-porosity, similar to the base 4340 steel, after the HDI demonstrating that the HDI processing almost completely eliminated the porosity in the plasma coating. Also note that the as sprayed, mechanically bonded interface between the coating and the base material was transformed into a metallurgical bond. Hardness profiling from the coating to the base material revealed that approximately 200 μm of the base material had been slightly over-tempered. Processing time was approximately 10 cm^2/s and the resultant hardness of the coating layer was 982 HV. Full thickness HDI fusion of plasma-sprayed coatings had only been successful with the nickel self fluxing alloy; therefore it was believed that a fundamental understanding of the HDI fusing process would allow for the process to fuse a wider range of thermally sprayed wear and corrosion resistant materials.

4.2.1 Initial Trials – Year 1

The initial scoping trials of various materials led to various results and conclusions about the HDI process.

Precursor Type / Summary of Results

Carbide / Cermet Suspensions

On the initial HDI tests coatings looked good from, 0.003 – 0.005”thick

Thermal Spray

- Stainless Steel: This alloy was flame sprayed, plasma sprayed and HVOF sprayed and initial HDI fusing studies showed that denser and less oxidized spray coatings resulted in significantly improved densification response and responded better to HDI treatments
- NiCrBSi / Self Fluxing: This alloy was also deposited via flame, plasma and HVOF, and all coatings densified well under a broad range of HDI conditions; therefore this alloy family was found not to be as sensitive to as sprayed coating structure as were other thermally sprayed precursor layer structures.
- Ni-cladding: Initial nickel claddings were deposited via plasma spraying and when HDI fused they showed complete lack of wetting and adhesion. Prelayers with added wetting “agent” such as Phosphorous, Boron and/or Silicon were found to aid the wetting and thus the precursor layer densification.
- Hastelloy C: Results were very similar to stainless steel thermally sprayed coatings.
- Tool Steels: similar behavior to the stainless steel coating.

The main aim of the initial testing in Year 1 had been to scope out the behavior of various sets of coating materials including tool steel powders and other carbides:metal matrix coatings and applied both as either thermally sprayed layers and as suspensions.

Ni Self Fluxing / Flame Spray Coatings

St Louis Metallizing had initially flame sprayed several of self fluxing Ni alloys (NiCrBSi) samples for HDI testing. They were HDI fused and coating results were very good. Dense and well adhered coatings were achieved... there was no dewetting/delamination of HDI processed coatings.

316 Stainless Steel / Flame Spray Coatings

After HDI processing, it was determined the coatings had too much oxidation, and HDI processing did not significantly densify the coating microstructure. These results led to the suspicion that too much oxide in the thermally sprayed precursor layers likely acted as insulating layers that prevented HDI densification. The surfaces of treated coupons were macroscopically very uneven (wavy appearance) due to a problem that thermal spray source (Saint Louis Metallizing, SLM) was having with controlling the correct spray layer overlap.

316 Stainless Steel / Flame Spray Coatings

Figure 4 show the microstructures of the cross section of the flame sprayed 316 stainless steel coatings. Figure 4a illustrates the as sprayed coating with significant particle boundary oxidation and debond. Figure 4b illustrates the microstructure of the coating after thermal fusion trials with the HDI process. One can see very little change occurred in the coating density and adherence level. In fact, HDI processing likely increased coating oxidation. These results were some of the initial results that showed the initial thermal spray structure was critical to HDI processing behavior and led to a narrowing of materials and scope that occurred in Year 2.

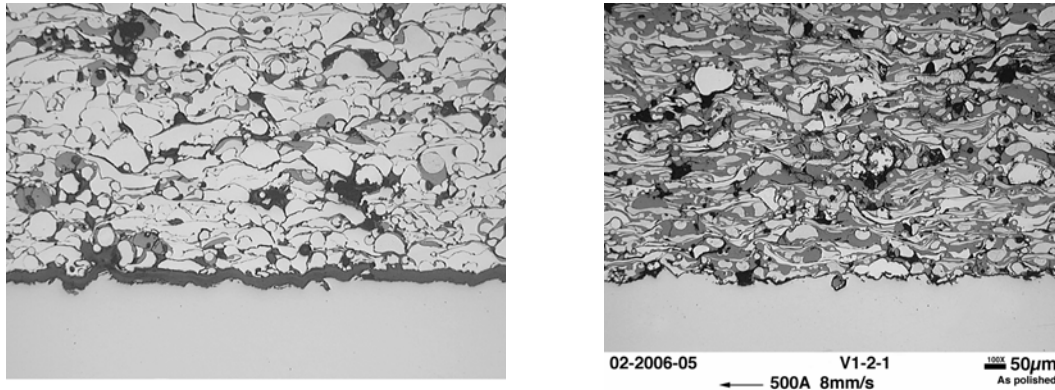


Figure 4. Flame Spray 316 SS onto Steel Substrate a) as-flame sprayed and b) after HDI thermal scanning at 500A and 8mm/sec.

316 Stainless Steel / Plasma Spray Coatings

Finer powder size distributions of 316 Stainless Steel powders were provided by Carpenter. These finer powders permitted better particle in the plasma jets and led to high density plasma sprayed coatings. However, the plasma sprayed precursor coatings still had variable porosity, oxidation and adherence, which led to mixed HDI processing results where some processed coatings almost fully densified when HDI fused, but they experienced significant delamination during fusion that led to non-uniform HDI fusion of the remaining portion of the plasma sprayed 316 stainless steel precursor layers.

316 Stainless Steel / HVOF Spray Coatings

This thermal spray process produced the densest and the most adhered as-sprayed coatings and also produced the best and most consistent HDI results, however additional samples were needed to optimize the HDI procedures that could yield full density on thick coatings ($> 0.020''$). HVOF coatings seemed to be more consistent as-sprayed, and therefore, responded more consistently to HDI processing/densification.

Hastelloy C / Plasma Spray Coatings

The same variability that plagued the plasma sprayed stainless steel coatings affected the poor outcome on the Hastelloy C precursor coatings. Variable coating, as-sprayed coating adhesion, led to delamination and variable HDI densification. This coating, as-sprayed, had a smoother surface finish than the arc-sprayed coating.

Hastelloy / Wire Arc

Wire arc spray was another type of thermal spray precursor coating that was evaluated. The arc sprayed Hastelloy coatings were very rough and not well bonded. The result was poor HDI densification, likely due to both the variable IR reflection of the rough surfaces and the poor thermal conductivity of porous coatings. This coating had a significant amount of gas entrapped from the spraying process.

Hastelloy / HVOF

As with the 316 Stainless Steel powders, the HVOF process coatings were densest and most adhered, thus the HDI process worked best. Although when fused, the HDI coatings did not completely wet the base steel coupons.

H13 Tool Steel / Plasma

–100 / + 325 mesh H13 tool steel powders were provided by Crucible to St. Louis Metallizing (SLM) for plasma spraying. Plasma sprayed surface was significantly rougher than the HVOF sprayed coatings. There was a significant amount of large pores as-sprayed and the precursor coating appeared bonded well in some samples; however, in other samples where the coating delaminated from the substrate, there was no bond. These inconsistent as-sprayed structures prevented full assessment of HDI processing.

H13 Tool Steel / HVOF

–325 mesh H13 tool steel powders were provided by Crucible to SLM for plasma spraying. These powders produced dense, low oxide, adherent as-sprayed coatings that also led to relatively good HDI coatings. This coating looked bonded to the substrate, with a smoother surface finish than the plasma sprayed coating.

Ni-Self Fluxing (NiCrBSi) / Flame Sprayed

These type coatings were used extensively by Lund International (project partner) for preparing coatings that were subsequently fused on continuous batch furnace fusing. NiCrBSi powders were sprayed as oxidized and porous layers onto steel substrates. Figure 5 illustrates the coating structures after HDI thermal scanning. The coating fused onto the steel coupons were smooth and well adhered as shown in Figure 5a which shows the overall coating appearance of the flame sprayed Ni-Self fluxing (NSF) coating surface after HDI treatment. The various microstructures of the HDI fused coating, shown in sequence in Figure 5, show various cross section microstructures of the HDI fused coating and illustrates that the coatings were well densified and generally well adhered.

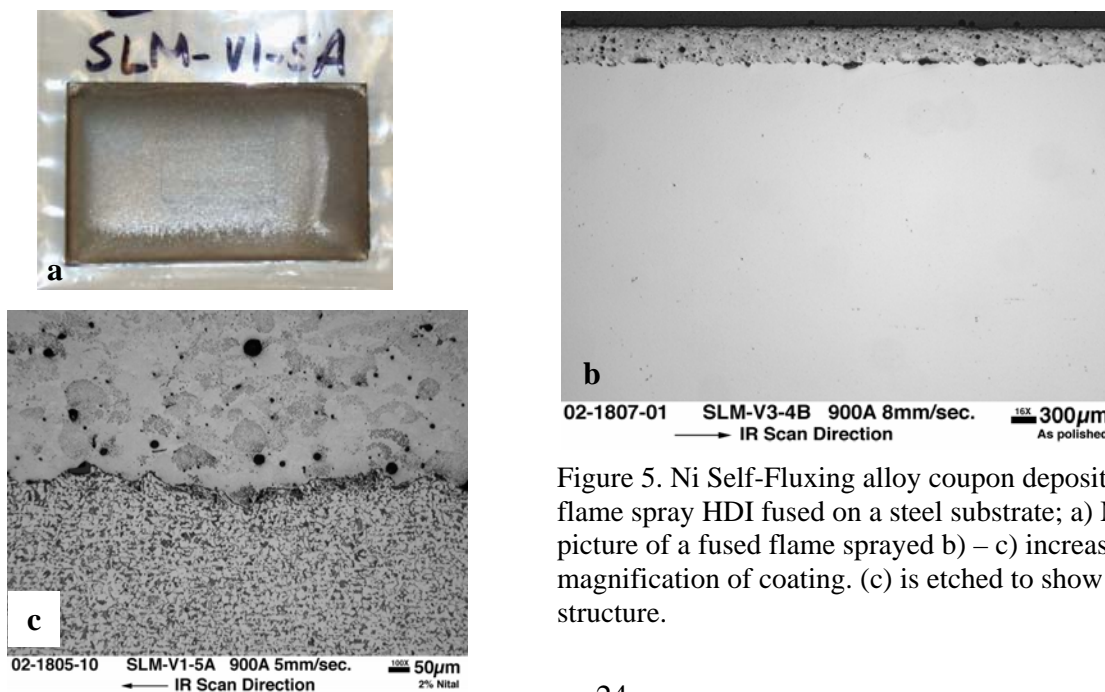


Figure 5. Ni Self-Fluxing alloy coupon deposited via flame spray HDI fused on a steel substrate; a) Macro picture of a fused flame sprayed b) – c) increasing magnification of coating. (c) is etched to show steel structure.

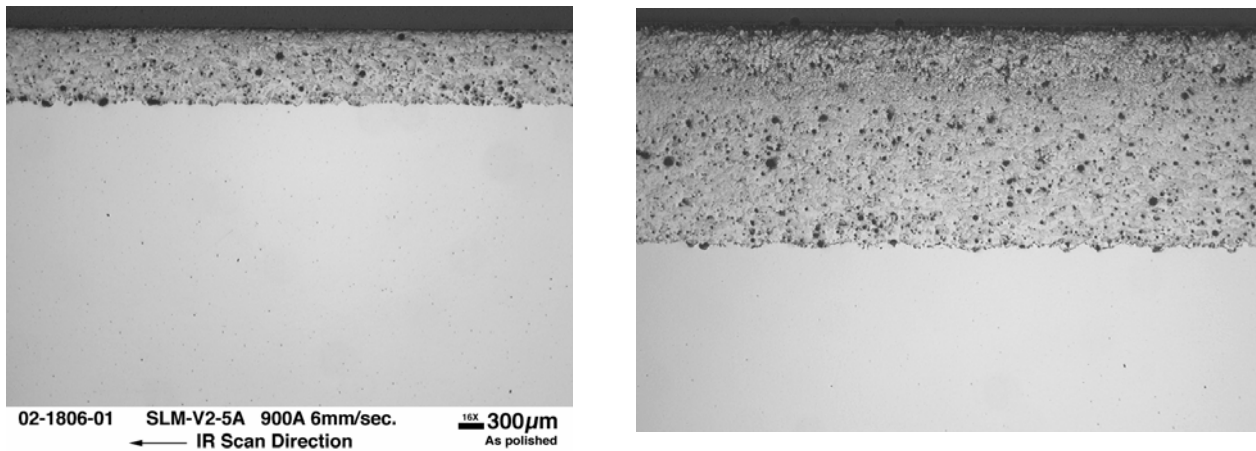


Figure 5(cont) Ni Self-Fluxing alloy coupon deposited via flame spray HDI fused on a steel substrate; d –e) increasing magnification of coating.

Ni Self-Fluxing (SF) coatings are used as wear coatings and is the main coating type for the agricultural blades manufactured by Lund International, one of the project's industrial partners. As such, the hardness of these coatings is a major factor in their performance. Hardness scans of the flame sprayed and HDI fused Ni SF coatings is shown in Figure 6. The hardness measurements illustrates that the HDI treatment has been successful in densifying the structure and made the coating more uniform over the entire coating thickness.

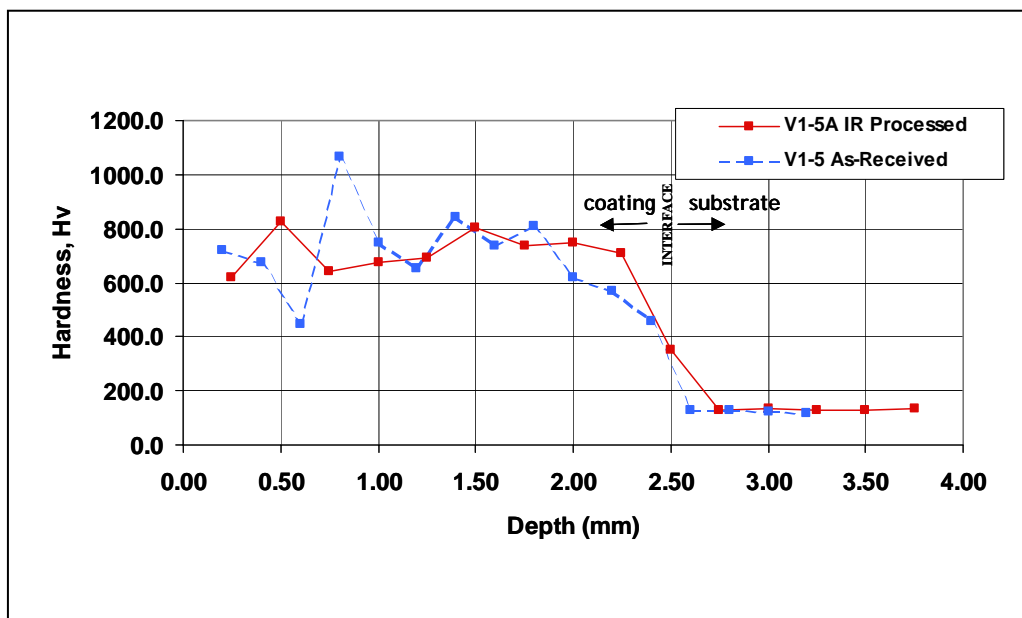


Figure 6. Hardness measurement scan on Ni Self-Fluxing alloy coupon deposited via flame spray HDI fused on a steel structure).

Nickel Coating Densification

As discussed before, the fusion of pure nickel coating that were HVOF sprayed onto steel coupons was not successful. Figure 7 is a picture of the attempt to surface fuse the HVOF sprayed Nickel layers. The bottom image shows how the nickel was melted and pooled the steel surface, not wetting the underlying steel substrate.



Figure 7. HVOF sprayed Ni layer on steel HDI fused with no wetting of the underlying steel substrate.

Tool Steel (CPM H13 & CPM S90V)

Figures 8 and 9 show the HVOF deposited precursor layers after HDI thermal scanning at the power and speeds indicated. One can see the original structures (spherical particles represent the initial powder structures) can be seen and that is was likely that little of no fusion occurred in the H13 tool steel and only some small degree of fusion occurred in the interfaces between particles, as seen in the etched structures. Figure 9 shows the structure of a different HVOF deposited tool steel, Crucible's CPM S90V that was also HDI treated. The general absence of the spherical powder particles may indicate better HVOF depositions, but more likely indicates that more fusion might have occurred in this particular precursor layer. Note that there is still little or no fusion through to the base steel, which can be seen in etched structure seen in Figure 9b.

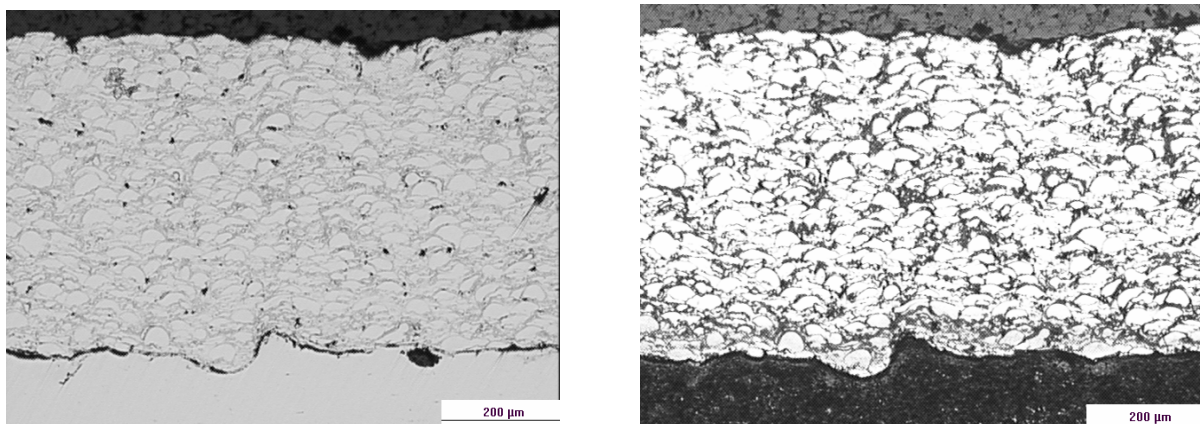


Figure 8. HVOF deposited CPM H13 tool steel HDI treated at 300A/10s+900A/2.5s; a) as polished and b) etched.

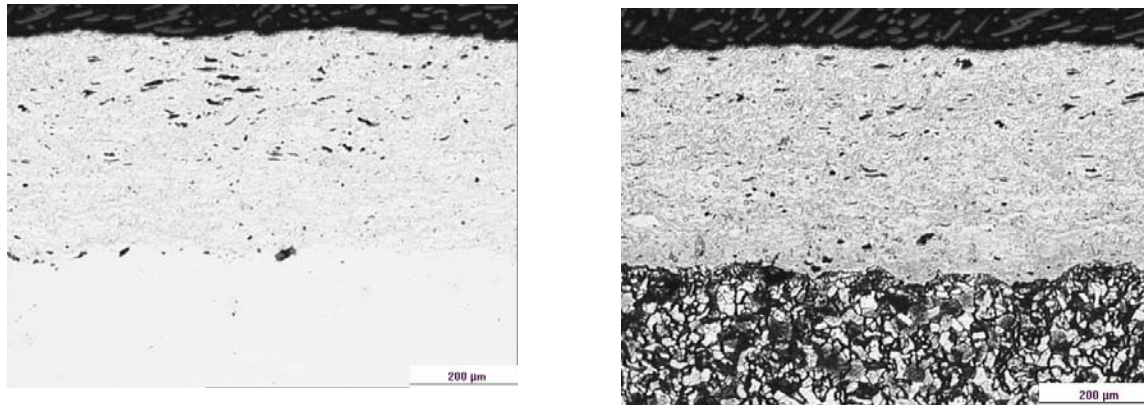


Figure 9. HVOF deposited CPM S90V tool steel that has been HDI treated; 750A/5s ; a) as polished and b) etched.

Figure 10 is a plot of the hardness values of the CPM S90V tool steel coating after HDI treatment. One can see that the coating hardness is quite high, but that there is a gradient in hardness in the base metal. This gradient is likely due to the heat treatment affects of the intense surface heating and the resultant martensitic transformation caused by the rapid quench related to the underlying bonded base materials that were not heated to the same degree. The gradient also indicates that it is unlikely that the coating was not completely fused since only the very outer layer of the coating had a hardness that exceeded R_c 60.

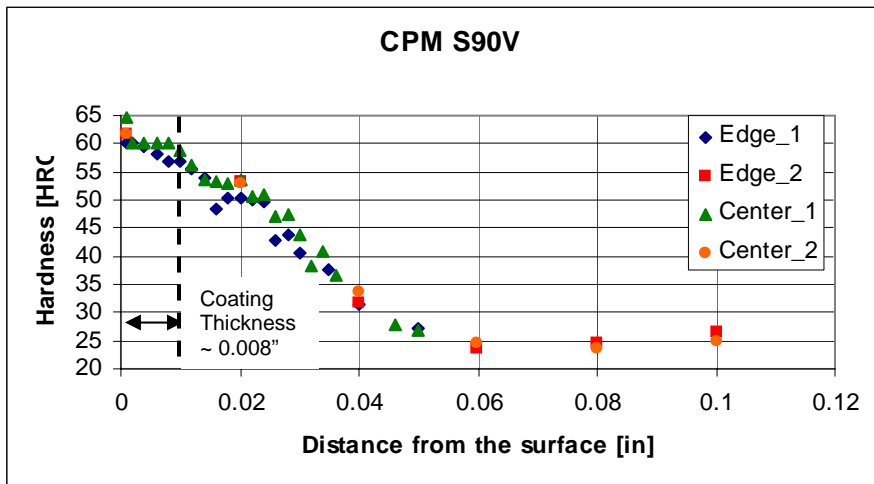


Figure 10. Hardness values of HVOF deposited CPM S90V tool steel that has been HDI treated.

Suspension / Cermet Coatings

The suspension coatings were a mixture of WC or Cr_3C_2 carbide particles mixed with a Ni-P or NiCr-P braze alloy powder in an organic binder. The binder was used to suspend, carry, and hold the particles onto surfaces before HDI treatment. These suspensions are normally sprayed, via compressed air, onto surfaces where they are dried to harden before fusion, either by furnace or in the case of this research by intense surface IR irradiation, termed HDI treated. The suspension coatings generally responded very well to densification, provided the precursor layers were less than 0.005" thick. Figure 11 shows the

microstructure of a HDI fused tungsten carbide, Ni-P cermet. The coating is fully fused, well bonded and dense indicating that suspension precursors with lower melting braze alloy particles responded well to HDI fusion treatments. It was also believed that the “black body” nature of the suspension layers increased the absorption of IR energies over the thermally sprayed precursors layers with little reflection.

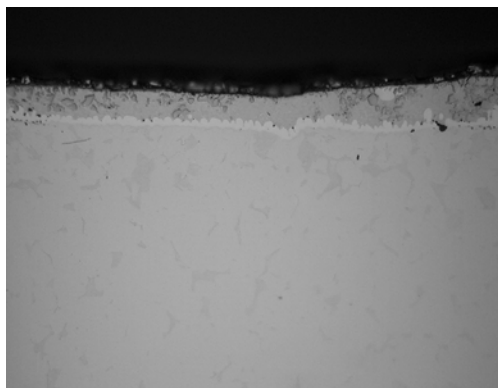


Figure 11. Cross-section of a tungsten carbide, Ni-P cermet directly fused to a 1040 steel substrate, 1.54 kW.cm² at 10 mm/sec.

Figure 12 and 13 show additional HDI fused suspension, cermet coatings where the Cr₃C₂ containing coating (Figure 12) is significantly less dense than the corresponding WC containing suspension precursor layer. These differences in HDI response were surprising and reinforced the need to understand how the basic thermal and physical properties of the precursor layers interact with the HDI radiation and with the subsequent fusion and densification behavior. One should note these coatings are ~ 0.002-.003” thick.

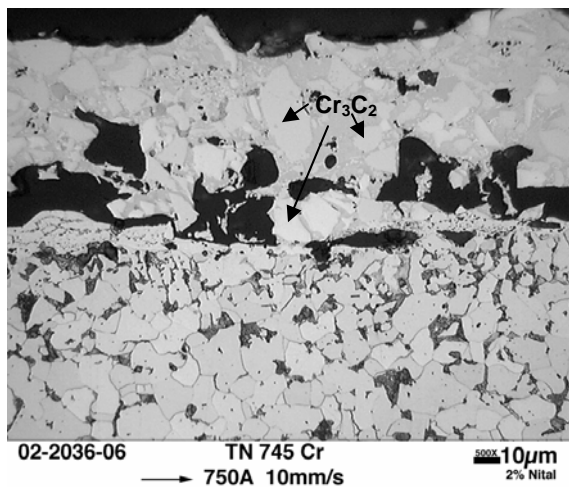
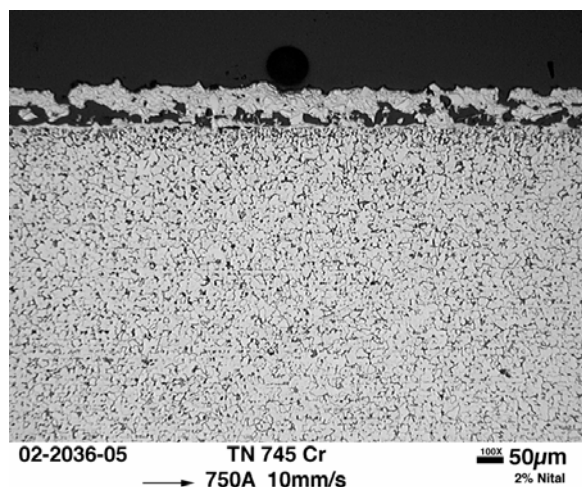


Figure 12. Cross-section of a chromium carbide(Cr₃C₂), NiC-P cermet directly fused to a 1040 steel substrate.

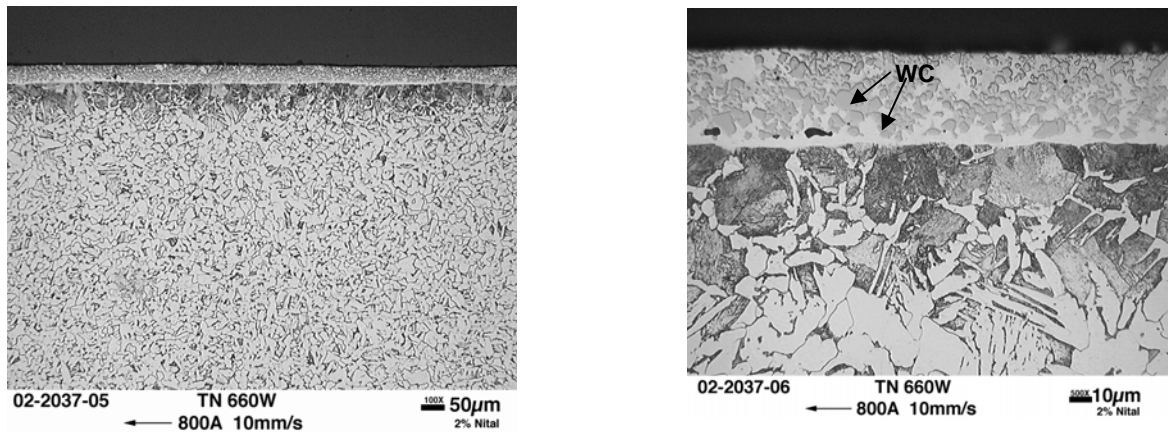


Figure 13. Cross-section of a tungsten carbide (WC), NiC-P cermet directly fused to a 1040 steel substrate.

Surface Hardening of Tool Steel

One of the industrial partners, Crucible, had interest in the bulk hardening of their tool steels and the HDI process was investigated for use in surface hardening the tool steel. Figure 14 is a plot of the hardness values of a CPM-10V tool steel, made from hot isostatically pressed powders. One can see that the tool steel was hardened to a depth of ~0.080" (2mm) using HDI surface scanning. This layer would likely provide for good abrasion resistance.

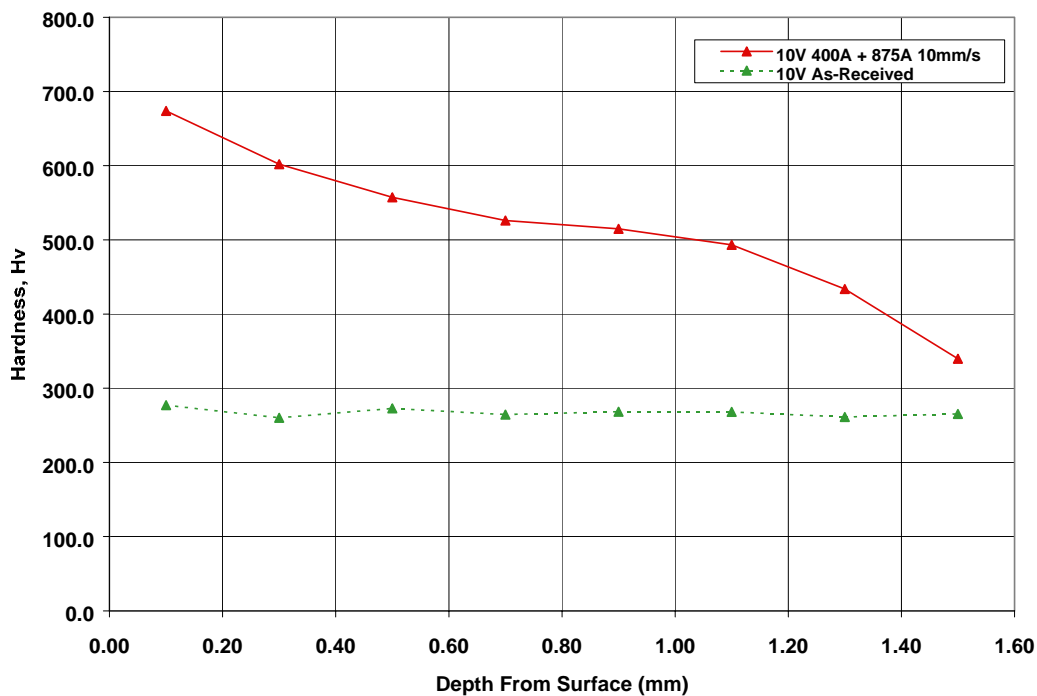


Figure 14. Cross-section hardness value profile of CPM-10V tool steel as HIP'd and after HDI treatment.

4.2.2 Initial Trials (Year 1) Conclusions

Thermal Spray Precursors

1. As-sprayed (flame, plasma, or HVOF) Self Fluxing alloys were found to be very tolerant precursor layers for HDI processing.
2. HVOF seems to be the best thermal spray coating process for alloys such as stainless steel, tool steel and Hastelloy. Their high density, low oxide content and good bonding promoted the best HDI response.
3. Plasma spray bonding was variable when spraying 316 Stainless and Hastelloy powders, leading to variable HDI fusion and densification.
4. There was a general “lack of wetting” when the fused metal layers did not contain B and/or Si.
5. Coating delamination of thermally sprayed 316 stainless steel layers occurred quite often and may be attributed to the rapid build up of CTE related stresses, which stress the interface, making the lower bond strength coatings (i.e, 316 Stainless Steel and Hastelloy) separate from the coated steel coupons.
6. HDI was found to be very sensitive to the variations in thermal spray coatings, potentially limiting thermal spray as a precursor layer method, with the exception of HVOF.

Suspension Precursor TN660W (NiP/WC)

Metallography showed these coatings to be dense and well adhered. These coatings were found to respond well to a range of HDI process parameters.

Suspensión Precursor TN745Cr (NiCrP/Cr₃C₂)

These coatings were not optimized. One hypothesis for why the TN745Cr coatings did not densify using similar HDI parameters to TN660W was that the Cr₃C₂ particles had lower thermal conductivity, thus requiring slower scan speeds and/or increased HDI heating power.

Suspension Precursor Tool Steel and/or TN745Cr - Mixtures

These suspensions consisted of either 100% tool steel powders or were a composite of the tool steel powders and the carbide powders, as the hard but tough reinforcing phase with a braze filler binder (either BNi-6 or BNi-7). Blends of the TN alloys with Rex 121 tool steel were sprayed and HDI processed. The initial indications were that there was mixed “wetting / coverage” of the fused suspension. It appeared that during HDI fusion, the lamp’s heating zone was pushing the generated molten layer out ahead of the heating zone.

When the tool steel powder CPM 10V was blended with carbide particles, the HDI densification/fusing behavior was variable. It appeared as if the tool steel powder particles were being melted in addition to the lower melting braze powders. It was thought that lower power or faster scan times be implemented so that only the braze binders and not the tool steel powders be fused. It had been hoped the HDI process would preferentially melt the braze powders as a binding fused phase, such that the tool steel powders would be encapsulated as still solid particles. Initial HDI results on these blends seemed to indicate that the braze bonder and the tool steel powders may be melting, causing the fluid layers to be moved by the thermal effects of the melting front as the IR beam moved across the coupon.

Conclusions on Suspensions:

In general, suspension coatings produced the best coatings, although the HDI process was variable and far from optimized. Some of the advantage could be related to the thinner nature of the suspension precursors..

1. TN660 W (with WC particles) was optimized for flat 2" x 3" coupons and were dense and well adhered.
2. TN745Cr still required optimization to achieve full density
3. Tool Steel blends with TN600 and TN700 become "too fluid" and do not uniformly coat the coupon surface. It is possible that the HDI power was too high and the tool steel particles were extensively remelted.

Conclusion on Nickel Cladding:

The objective, as requested by Ametek, was to see if HDI could produce clad nickel sheet and eventually replace the welding/rolling processes used now by Ametek.

Initial trials were unsuccessful as the nickel layer upon melting pooled up and did not wet the underlying steel layer. Two issues were suspected. First, a chemical wetting layer was needed. Elements such as B, Si, P, and Cr were seen to promote wetting reactions on steel. It was suggested that underlayers of B and/or Si should be added that would promote wetting. Ni-with TN200 (NiCrBSi) underlayer (contained self fluxing) was tried but initial experiments indicated similar 'lack of wetting' of the base steel upon fusion

Year 1 concluded with the recommendation that fewer material compositions be studied and more focus be placed on developing an HDI process model which was based on thermal-physical properties of the precursor layers. The next section(s) describes the approach and the results.

4.3 HDI Process Studies and Modeling-Years 2-4

The Initial Studies; Year 1 work cited the following technical issues: 1.) Inconsistent melting of the thermally sprayed precursor layers; 2.) Lack of wetting of the steel substrates by the molten layers; and 3.) Inconsistent HDI fusion and densification of coatings. The revised, Years 2-4 experimental plan, had the following fundamental elements to address these issues and better understand the HDI process:

HDI / Coating-Substrate Thermal Modeling

- Measurements of precursor layer thermal properties (C_p [ratio of specific heats], k [thermal conductivity], α [thermal diffusivity] , and H_f [heat of fusion])
- Evaluation of thermal spray coating structure and properties
- Compute thermal profile calculations based on properties

Density / bonding

- Characterization of Post HDI Layers
 - Bonding
 - Structure
- Focus on Fewer Material Systems (precursor / substrate)
 - Ni-CrBSi Flame Spray Coating
 - Stainless Steel HVOF (high velocity oxy-fuel) spray coating
 - Tool Steel (CPM) by HVOF
 - Suspensions with Carbide and/or Tool Steel reinforcements

The technical aspects of the revised project plan were set to select “model” material systems (precursor/substrate) including: Ni-CrBSi Flame Spray Coating, Stainless Steel HVOF (high velocity oxy-fuel) spray coating, and Tool Steel (CPM) by HVOF, and Suspensions with Carbide and/or Tool Steel reinforcements. In addition to these basic studies, the project was to center on Lund Agricultural Cutting Blades as the industrial application. Lund International, a project partner, contributed 75 flame sprayed agricultural blades for HDI fusion tests.

As discussed above, a more fundamental and basic approach was designed to understanding how the HDI process interacts with precursor layers and substrates to form wear coatings. An experimental plan was devised to address and better understand the influence of the coating/substrate material properties on bonding characteristics. Figure 15 illustrates this experimental plan that carried the investigation through Years 2-4.

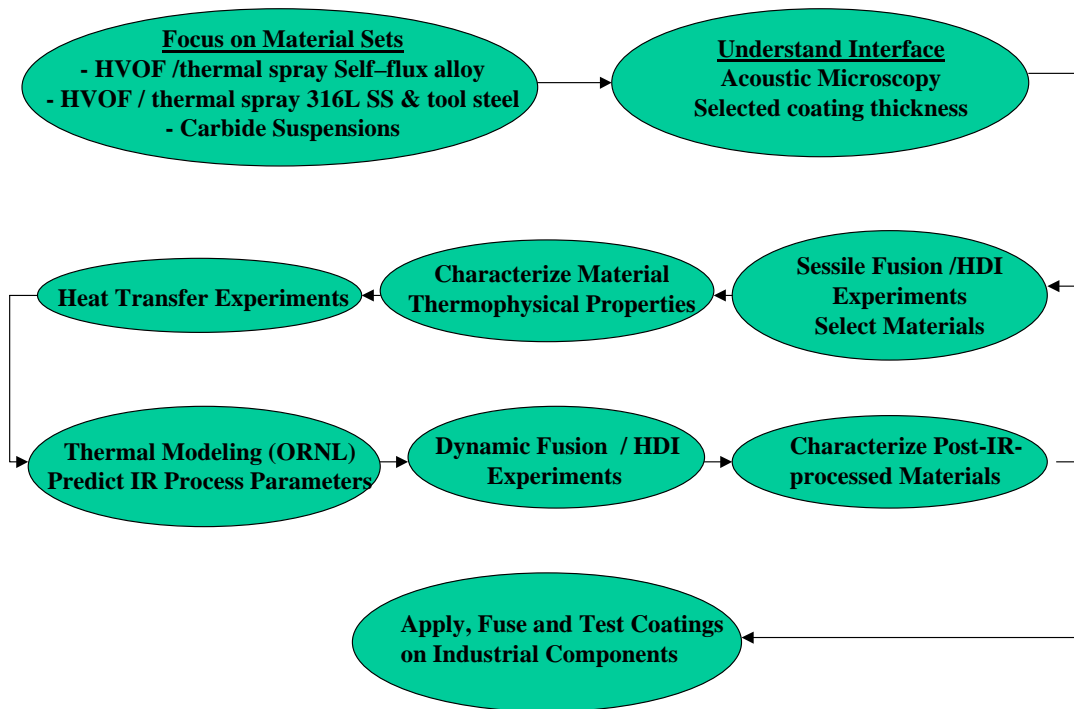


Figure 15. Schematic of Fundamental studies experimental approach.

4.4 Thermal Modeling

4.4.1 Model Development and Experimentation

Thermal modeling of the pulsed thermal profile of the HDI lamp / process, shown in Figure 16, was used as the base for all subsequent thermal modeling. In this model, the incident energy is shown to be absorbed more by the solid coating precursor layer, where as, after melting, the emmitance of the molten pool changes and very little of the incident energy is absorbed.

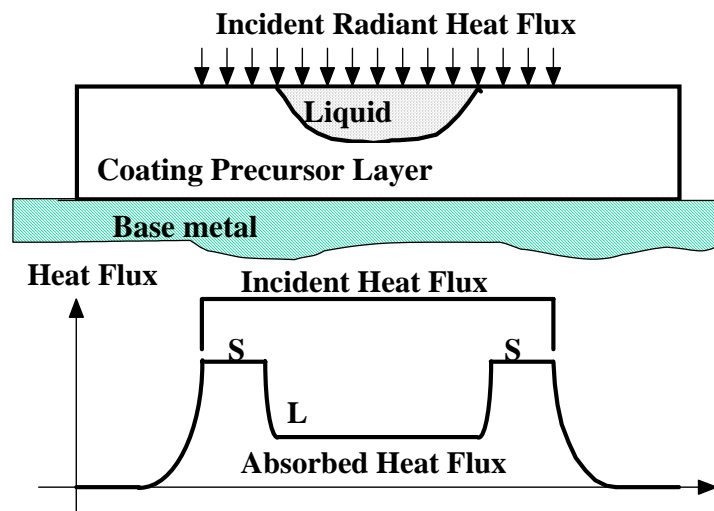


Figure 16. Illustration of thermal model as coating layers are melted by the incident IR beam.

Figure 17 illustrates the coating layer model for the HDI fusion process. Input parameters were for pure nickel on steel. Figure 17a depicts the prediction of the temperature profile on the coated surface during a sessile / pulse of the HDI lamp that had a pre-pulse at 300A for 10 seconds followed by a 900 A for 2 second pulse. The “sessile pulse” means that the lamp is held stationary and irradiates the exposed surface area of the clad surface. Figure 17b depicts a liquid containing, based on the thermal properties of a pure nickel layer. Figure 17 illustrates that the thermal model for the HDI process how the modeling effort advanced the understanding of IR heating and surface fusion. The modeling effort included collecting the thermal property data on the actual HDI coating precursors selected for Year 2-4 investigations (see Figure 15).

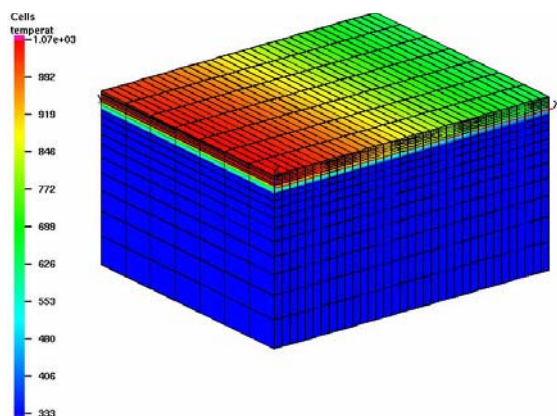


Figure 17a. Temperature profile from HDI Model

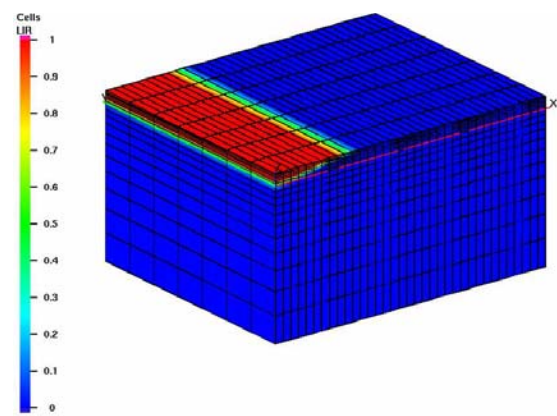


Figure 17b. % Liquid in Coating from model

Figure 18 is a photograph of one of the flame sprayed, self fluxing alloy coated steel test coupons which had been subjected to the sessile pulse, described above. One can note the fused region of the coated surface and the profile of the fused regions.

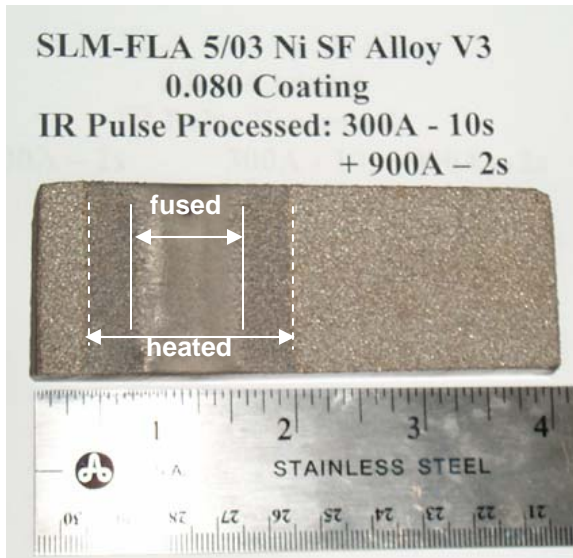


Figure 18. Macro photograph of coated coupons
Flame sprayed Ni-Self fluxing alloy
precursor coating. Pulse IR treated.

The experiments that followed these initial model developments included the processing of instrumented sessile / impulse HDI fusion tests and the analysis of thermal-physical property specimens that included the stainless steel, the self-fluxing alloy, the tool steel and the suspension particulates. Tests were conducted on the various coating materials. Their thermal property data were then used to supplement the HDI fusion model whose output is shown in Figure 17. During these basic modeling and test experiments, ORNL continued to develop the HDI process parameters and gain processing experience on industrial components based upon Lund International's agricultural blades.

Figures 19 and 20 are photomicrographs of the HVOF sprayed CPM 90 V tool steel coating and the Ni-SelfFluxing coatings treated with differing HDI processing techniques. They show an improvement of fusing that occurred from the knowledge gained from thermal modeling and the implementation of the heat soak pass.

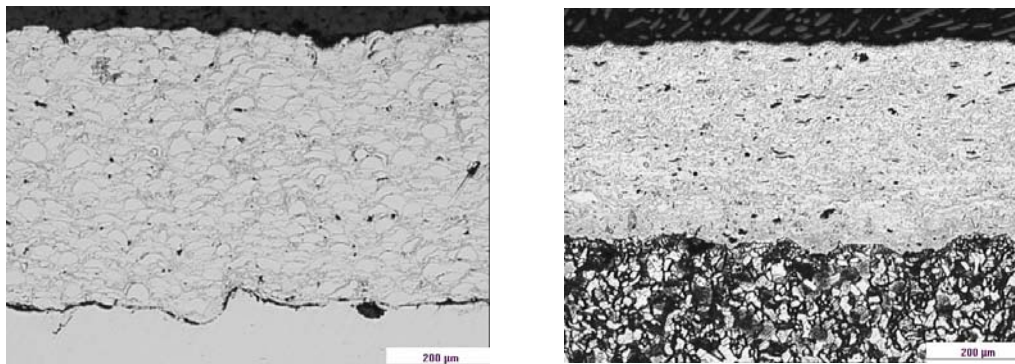


Figure 19. HVOF deposited CPM S90V tool steel that has been HDI treated; 750A/5s; a) as polished and b) etched. (repeated from Figure 9)

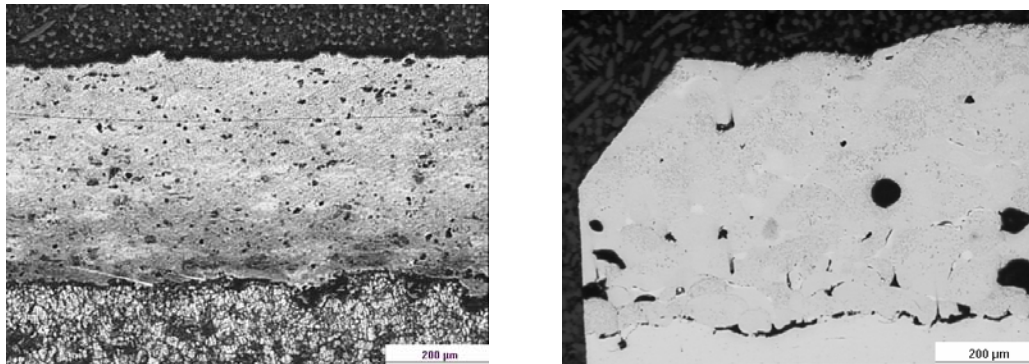


Figure 20. HVOF deposited NiCrBSi (Ni-Self Fluxing) coating that has been HDI treated; 300A / 5s followed by 850A/5s ; a) as polished and b) etched.

These figures show the varying degree to which the infrared irradiation densified the thermally sprayed layers of tool steel and NiCrSiB type self fluxing coatings. These layers were made by first depositing the materials (from powders) onto a 3" x 3" steel coupons using a High Velocity Oxy-Fuel (HVOF) process or in the case of the self fluxing Ni-alloy, a standard combustion flame spray process. These structures were used as feed back to the thermal modeling to assist in predicting the degree of melting based on first principles using material thermal properties and on heating the IR heating model.

The following samples were made and tested:

Thermal Diffusivity – Suspensions: TN745Cr, TN700, TN660W, TN745Cr+32REX, and TN32CPM10V
Bulk Powders: HIP Tool Steel samples (except for CPM 10V and H13). HVOF
CPM 90V, CPM 10V, and H13 samples

Thermal Conductivity: from the same set of prepared materials

Heat Capacity- Suspensions: TN745Cr, TN700, TN660W, TN745Cr+REX121, and TN745 +CPM10V
TN745Cr, TN700,& TN745Cr+32REX, HIP CPM 90V, H13, and S10V -

The conclusion of this work created a thermal property data base for the materials being studied. The table in Appendix 1 presents the final thermal properties that were determined by the various measurements. Based on these measured properties, ORNL conducted thermal modeling in order to predict IR Process Parameters. From the modeling, it was learned that lower power thermal scans to preheat the base behind the surface layers was important for more uniform surface heating and fusion. The results of HDI fusing experiments that relied on this technique are presented below.

Basic experiments were completed on two (2) TN + tool steel particle suspension mixes to determine the actual melt temperatures of these multi-alloy mixes, using previously determined thermo-physical property data for these materials. These results were utilized to establish the initial experiments needed to determine the IR thermal processing parameters for these complex materials. Kinetic (as opposed to the static pulse heating) Heat Flux Experiments were completed for these two TN + tool steel particle suspension mixes. The model relied on a 2-layer model that enabled the prediction of kinetic (dynamic scanning) IR processing parameters for these coating-substrate combinations. Figure 21, highlights some

of the predictive capabilities of ORNL's HDI process model to determine coupon surface temperatures and state of coating melting.

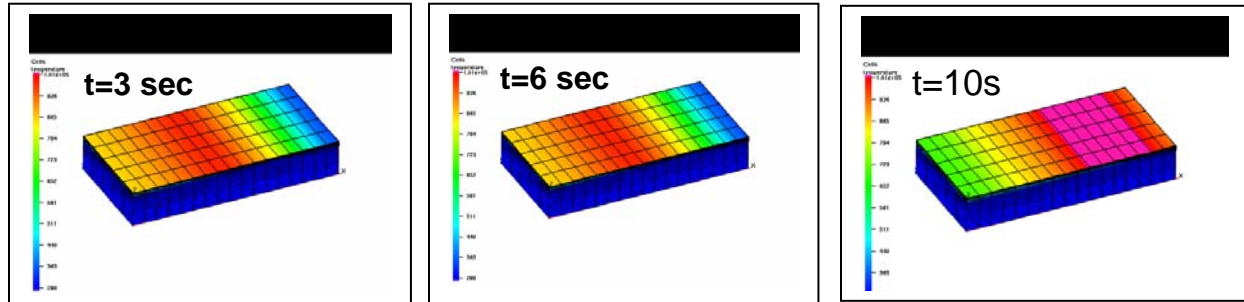


Figure 21. Thermal Modeling Simulation of Temperature Distribution During Kinetic IR Processing
TN745Cr + 10V + ...Suspension Coating 900A@ 6mm/s

Several application-focused material coating/substrate combinations were thermally sprayed and samples were instrumented to determine the actual melt temperatures for these sprayed materials. These experiments helped define the critical temperature regime for the coating that needed to be maintained to guarantee optimal matrix fusion while leaving the carbides from dissolving. The dissolution of carbides would likely have reduced the coatings' "fused" wear performance

Experimental work and HDI process development investigations were conducted to develop HDI processing methods for suspension precursor coatings based on the model predictions. This work led to significantly improved carbide / tool steel composite coatings as shown in Figures 22 and 23. Both figures show that over 98% dense and well adhered coatings were able to be produced at thicknesses of 200 μ m. This development led to a much more optimized coating structure from which wear test samples were made. The coatings show uniform, well dispersed composite (Cr_3C_2 , tool steel, NiCr matrix) coatings that are well fused and adhered to the base steel.

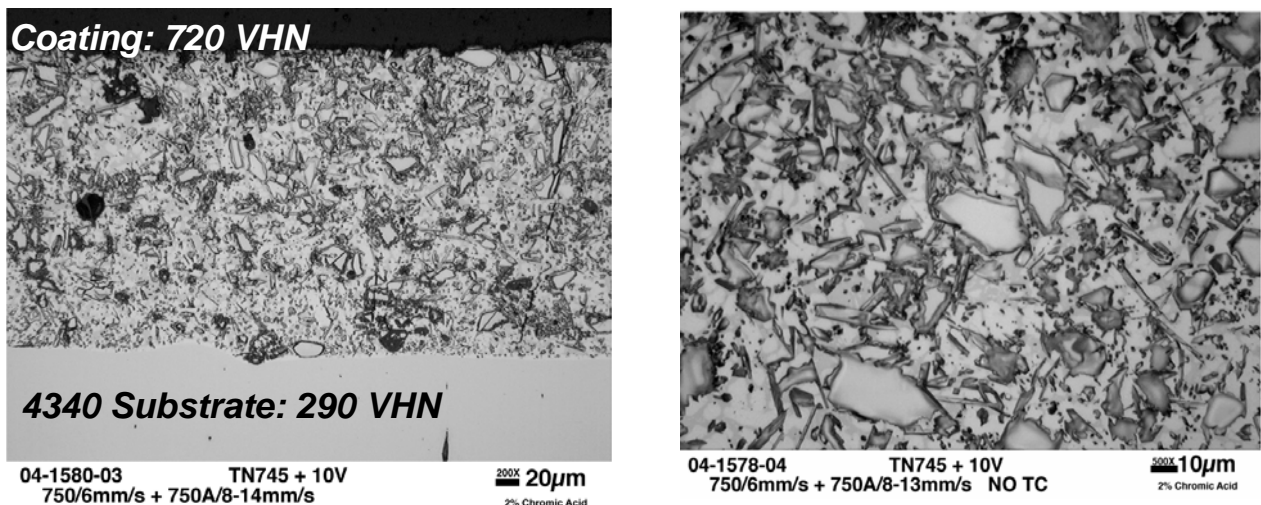


Figure 22. Photomicrograph of TN745 ($\text{Cr}_3\text{C}_2/\text{NiCrP}$) with CPM 10V tool steel suspension coating HDI treated using 750A traversing at 6 mm/sec followed by 750A with accelerated traversing from 8 – 14 mm/sec.

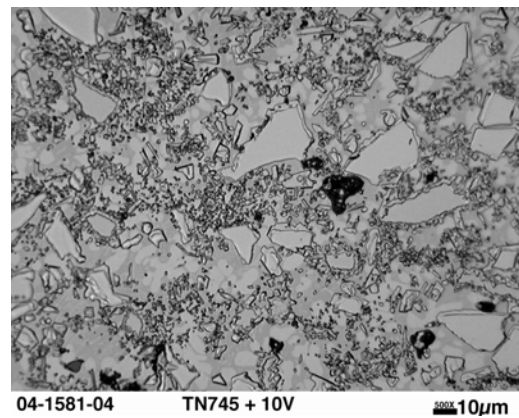
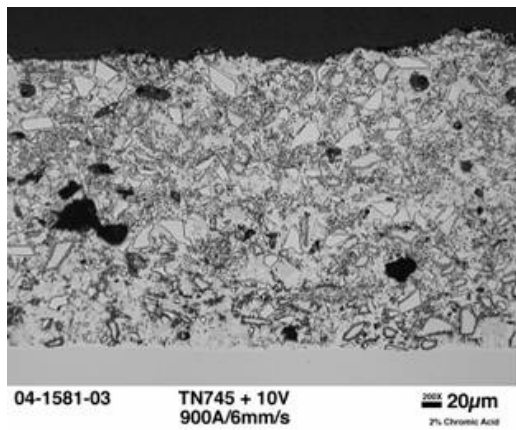


Figure 23). Photomicrograph of TN745 ($\text{Cr}_3\text{C}_2/\text{NiCrP}$) with CPM 10V tool steel suspension coating HDI treated using 950A, traversing at 6mm/sec.

The hardness of these HDI processed composite suspension coatings, mixing $\text{NiCrP-Cr}_3\text{C}_2$ with the CPM 10V particles, are shown in Figure 24 and compares the peak hardness of the tool steel modified coating to a WC particle / NiCrP (TN660W) HDI processed composite suspension coating. The much harder coating composite with the CPM10V tool steel particles illustrates the coatings were well fused and that the rapid quench from the fused condition initiated the martensitic transformation in the tool steel component of the coating. The innovation with the addition of tool steels to the $\text{NiCrP}+\text{Cr}_3\text{C}_2$ coating thus indicated that significant improvements in wear resistance could be realized.

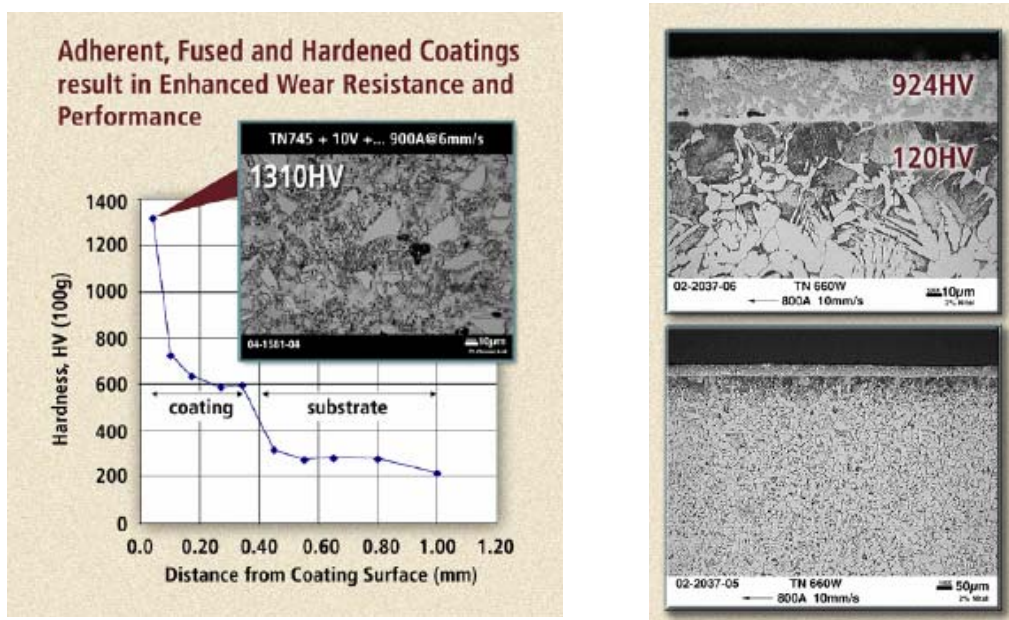


Figure 24. Photomicrograph / hardness comparisons of TN745 ($\text{Cr}_3\text{C}_2/\text{NiCrP}$) with CPM 10V tool steel suspension coating to TN660W composite coatings.

4.4.2 Modeling Results

A thermal flux model was developed that predicted the heat flux input & fusing processing parameters for wear resistant coatings. Excellent agreement was demonstrated between experimental thermal measurements (from thermocouple instrumented panels) and predicted thermal response for the coating cases modeled on the Ni Self-Fluxing and TN suspension coatings. These results shown in Figure 25 and 26 show the model worked quite well for the selected suspension coatings.

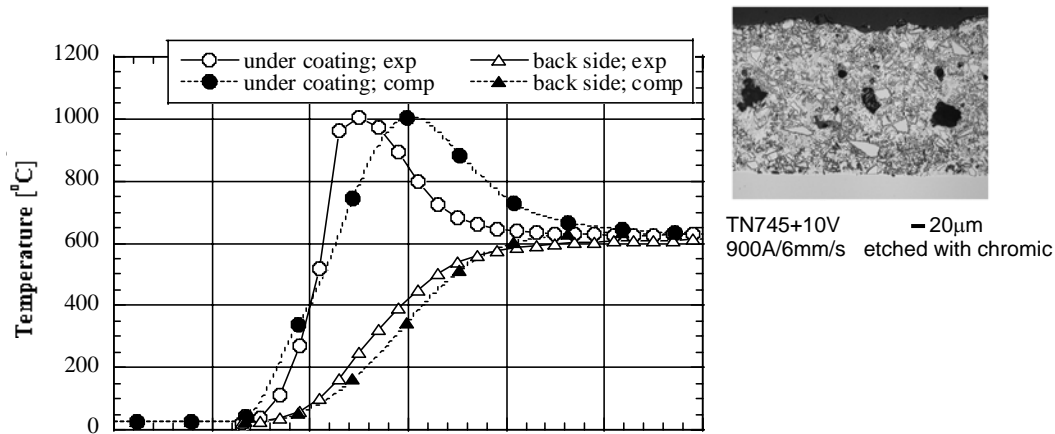


Figure 25. Thermal Profiles that were experimentally measured vs. those that were predicted by the model for a TN Suspension Coating demonstrate excellent agreement.

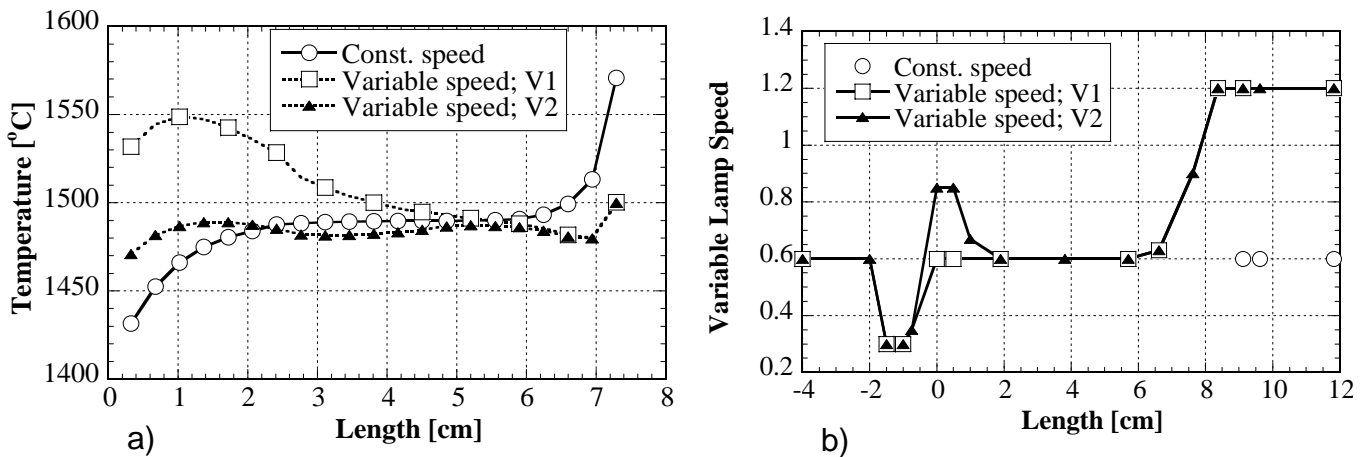


Figure 26. Model Thermal profiles; a) shows Predicted temperature across a 3 inch wide sample as a function of scan speeds

4.4.3 Baseline Thermal Trials to Establish Initial HDI Processing Parameters

Baseline thermal trials were initiated in order to establish the HDI processing parameters needed to melt the matrix material of the cermet based suspension coatings, without melting and putting the hard particles into solution within the suspensions' matrix material. The temperatures for thermal processing were based on the liquidus and solidus temperatures that were determined for the suspension coatings using differential thermal analyses (DTA). The metallographic and hardness evaluation of samples processed within these temperature ranges were then used as a guide to determine the thermal input needed to achieve these temperatures. These results were utilized in conjunction with instrumented HDI samples during HDI processing trials to establish coupon data on samples with model predictions. Experimental trials in conjunction with modeling simulations aided in establishing the HDI processing parameters needed to achieve the desired microstructures and hardness values. Figures 27 through 28 demonstrate the successful results obtained using the HDI processing parameters recommended by the processing models. Coating hardness values of up to 1310 H_v were achieved where the substrate hardness was only ~290 Hv. Figure 27 shows the complete, homogeneous densification of a suspension coating over a 3" x 3" surface. The macro-photograph shows excellent surface finish and the microstructure is dense, well fused and well bonded.

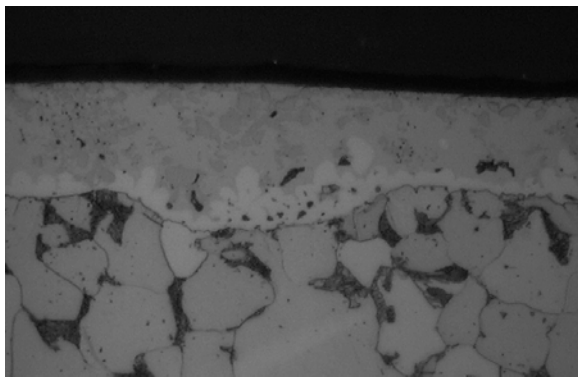
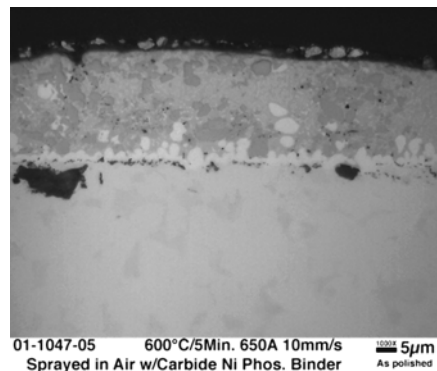


Figure 27 . WC/Ni- suspension on a steel coupon that was first air sprayed then HDI fused at 1.54 kW/cm² and 10 mm/s and the corresponding metallurgical cross-section.

Figures 28 – 31 show additional metallurgical and hardness results on HDI treated suspension-composite coatings. As with the WC/NiP coatings, they are well fused, dense and adherent.

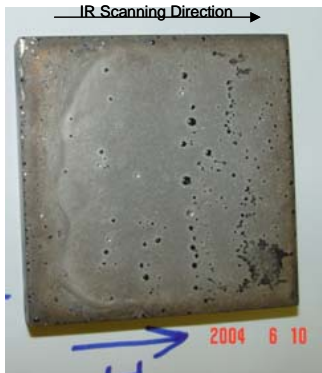


Figure 28. HDI Processed TN745Cr + 10PM 10V coating on 4340 type steel 3" x 3" x 0.25" substrates.

750A @6mm/s + 750@ 8-13mm/s, Ar purge

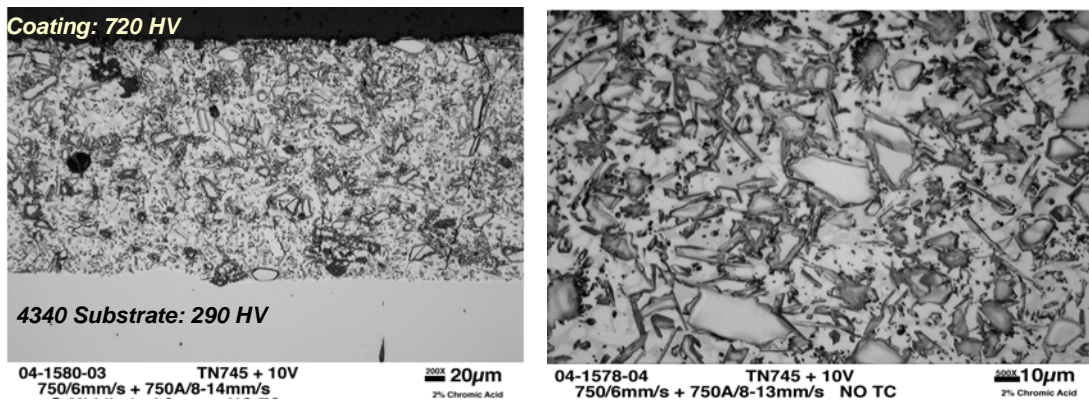


Figure 29. Photomicrograph of TN745 ($\text{Cr}_3\text{C}_2/\text{NiCrP}$) with CPM 10V tool steel suspension coating HDI treated using 750A traversing at 6 mm/sec followed by 750A with accelerated traversing from 8 – 14 mm/sec.

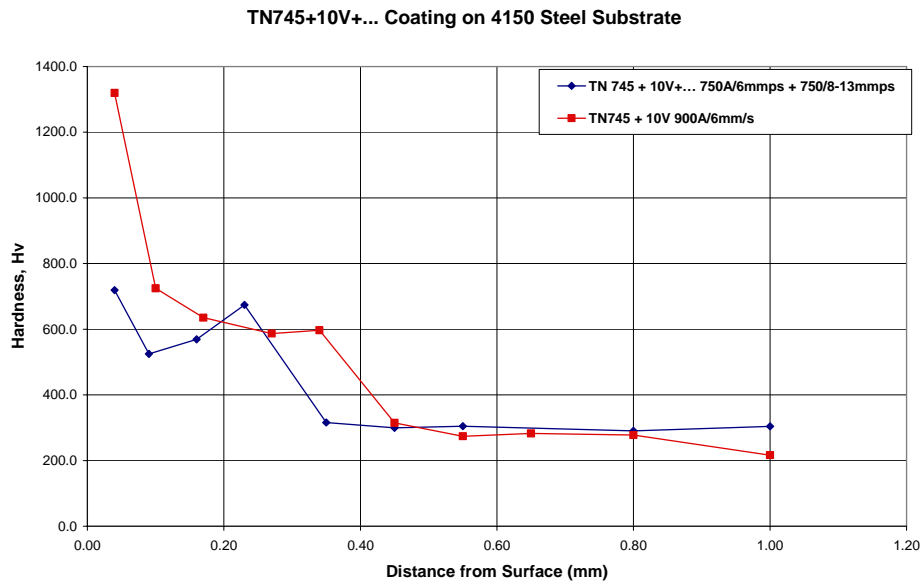


Figure 30. Hardness Profile Comparisons for two step IR Processing of TN745 ($\text{Cr}_3\text{C}_2/\text{NiCrP}$) with CPM 10V tool steel suspension coating HDI treated using 750A traversing at 6 mm/sec followed by 800A with accelerated traversing from 8 – 14 mm/sec.

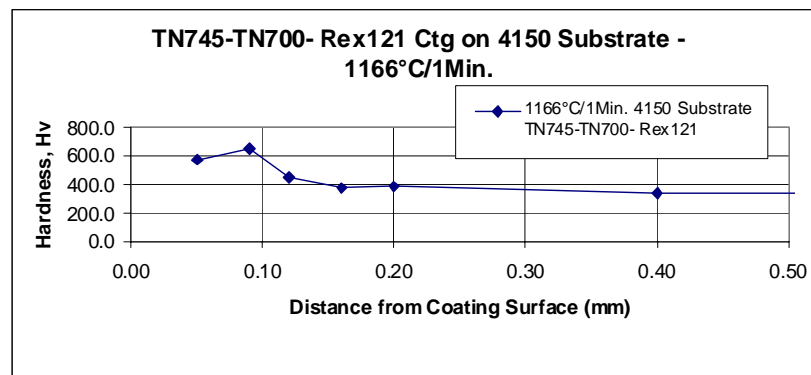
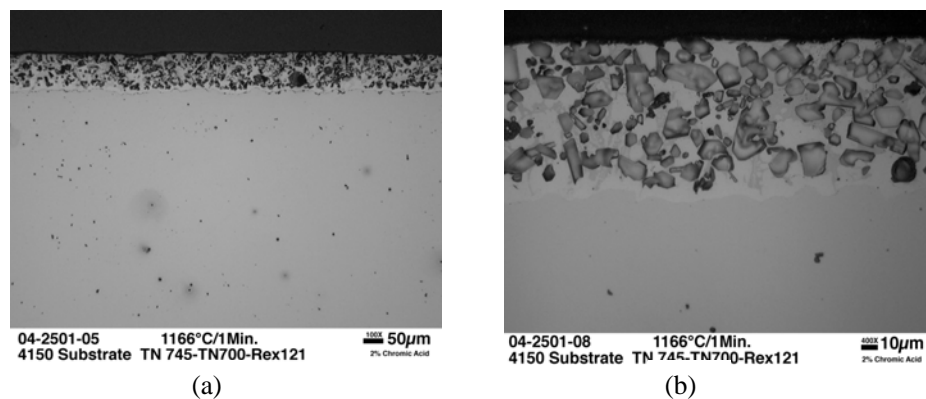


Figure 31. HDI processed composite TN700+ TN745Cr +Rex121 coatings; structure and hardness profiles.

Figure 31 illustrates another composite mixture with a “super” hard tool steel powder from Crucible, called Rex 121. It was also mixed with an excess of NiCrP to ease the densification using the wider melting range braze alloy composition. The structure shows much more equiaxed carbides but is also seen to be dense and well adhered. The modeling showed that preheating and accelerating the scanning through out the pass, to prevent overheating of the coatings as the IR lamp approached the edge of the specimens, led to excellent coatings. With these results it was determined that the HDI process was sufficiently understood and controlled to enter the final coating testing phase.

4.4 Coating Test and Evaluations

ORNL made HDI fused coatings onto the standard 3” x 3” x ½” steel coupons for wear testing. Figure 32 shows two of the coated wear test specimen blanks were coated using the most optimum HDI condition on the suspension cermet and composite with tool steel coatings. The coating surfaces were relatively smooth and uniform. Note that the darker spots were found to be oxides that have floated onto the coated surfaces during fusion.

The ORNL-HDI facility was used to fuse suspensions TN745Cr, TN745Cr plus CPV10, TN 745Cr plus Rex 121 for use in wear testing. Additionally, Lund International prepared 3”x 3” steel coupons and flame spray coated NiCr-Self Fluxing coatings which were also HDI treated at ORNL. The aim was to characterize the wear resistance of the HDI fused cermet and composite suspension as well as the

thermally sprayed (flame sprayed) Ni-Self-Fluxing alloy coatings that are used as the base coating by Lund for their agricultural blades. Wear testing was to be conducted under three modes. Sand abrasion, abrasive testing and sliding wear. The following section summarizes the wear results of various HDI processed precursor layers.

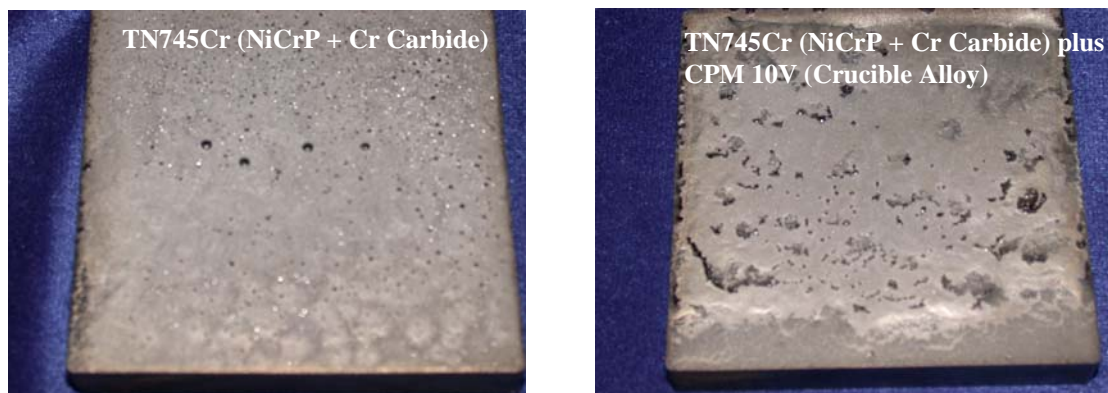


Figure 32. HDI fused suspension coatings on 3" x 3" x 1/2" steel coupons to be used as wear test blanks.

4.4.1 Sand Rubber Wheel (SRW) and Pin Abrasion Wear testing

Sand rubber wheel (SRW) testing was conducted at Climax Research in accordance with ASTM G-65 Procedure A, 30 lbs force and 6000 revolutions. Weight loss results are summarized in Table 1.

Table 1 Summary of Sand Rubber Wheel Test of Coated Coupons (mg. weigh loss of specimens)

	1	2	3	4	5	8	9	10	11	12	13
	HDI Fused 745	HDI Fused 745+10V	HDI Fused 745+Rex121	Tunnel Fused FSNi SF	FSNi-SF (HDI)	Vac. Fused 745	Vac. Fused 745+10V	Vac. Fused 745+ Rex121	Vac. Fused BrazeCoat C	Vac.Fused BrazeCoat W	Vac. Fused BrazeCoat CW3
Non-Ground	148	162	125	256*	193	134	176	276			
	178*	616*	158*		206*	153	184	263			
	161*	676*	155*								
Ground	1557	1684			188	383	899	1368	128	68	77
	1445	420			192	576	936	1411	133	58	
					217	761	993	1654			

The wear loss data in Table 1 shows that for the HDI processed specimens, the unground specimens had the lowest weight losses, while the ground specimens had very significant weight losses. This is believed to have been the result of the HDI processed coating being very thin and it was found that the grinding procedures removed most of the coating. The least weight loss (most resistant to wear) was on the HDI processed coating TN745 + Rex 121 coating. This coating was a blended suspension of NiP braze with Cr-carbide and the Crucible tool steel Rex 121 powder particles. There was significant variation with the TN745 + 10V which may be attributed to the higher wear samples having too thin a coating.

The tunnel fused flame sprayed (FS) Nickel Self-Fluxing (NiSF) coating, a standard industry (Lund) coating, showed to have about 40% higher weight loss than the TN745 + Rex 121 coating , which is

consistent with the hardness data. This table also compares the HDI fused coatings (ORNL) to standard vacuum furnace fused suspension coatings and also compares the suspension coatings (~ 0.006") to the standard "ground" BrazeCoat® thick (0.060") mat based coatings made with Cr-carbide (C) , tungsten-carbide (WC) and mix of Cr-carbide with WC. The BrazeCoat® coatings were made from placed braze infiltrated mats that had ~ 60v/o Cr-carbide while the TN 745 type suspensions typically had ~ 50v/o Cr-carbide. The SRW tests showed the BrazeCoat® coatings to consistently have the lowest weight losses, with the Cr-carbide BrazeCoat® coating (C) having very similar wear loss to the unground HDI fused TN745 coating and the vacuum furnace fused TN 745 coating. This showed that that HDI fusion in a protective gas shroud for both the TN745 and TN745 + 10V coatings, yielded near equivalent low wear rates to vacuum furnace fused coatings.

Figure 33 summarizes results in Table 1. Note that for the As-HDI processed coatings, the TN745Cr (NiCrP plus Cr Carbide) + Rex 121 (tool steel from Crucible) composite HDI processed coating had the lowest wear loss. But if the coating was ground, the wear rate was very high. It was later confirmed that the coating had been very thin due to the grinding process and did not represent the actual wear rate. The other comparison is the HDI coatings to the furnace used coatings. The wear rate of the HDI processed coatings are generally 50-75% of that of the vacuum fused coatings, indicating that the HDI process is at least as good as the vacuum furnace fusing for the suspensions and the flame sprayed coating. This finding confirms the utility of the HDI process over vacuum furnace treating. BC-W coatings that are furnace fused very high carbide content coatings are also shown for comparison and the HDI coatings fare well, except that the BC-W coatings are 10x the thickness of the HDI coatings.

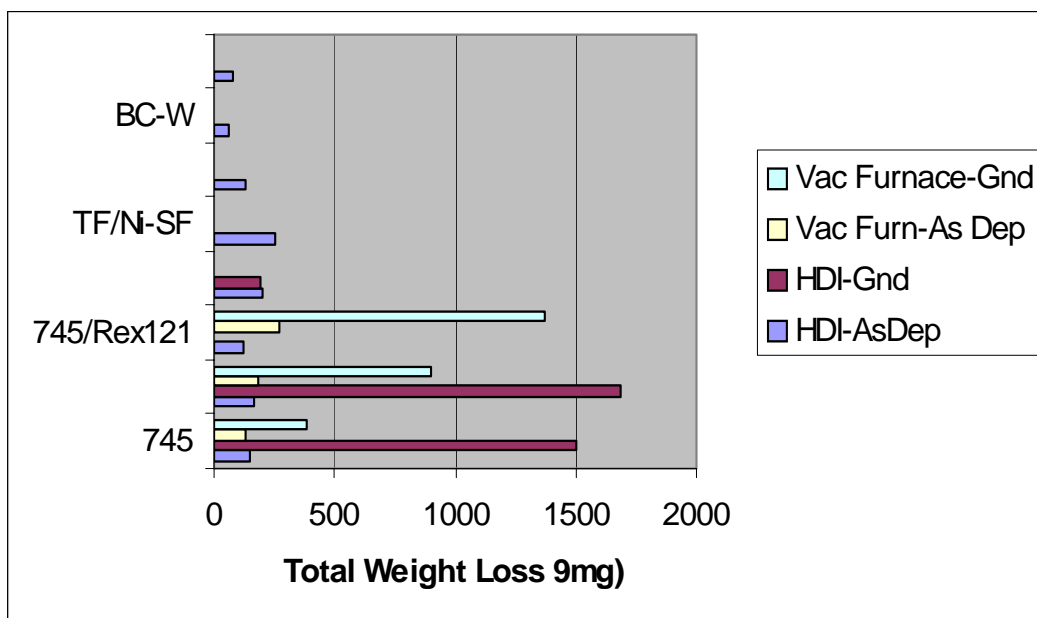


Figure 33. Chart of Sand Rubber Wheel abrasion test results.

The HDI fusion coating that really showed an advantage was the TN745 + Rex 121 coating. It was likely that the rapid heating and cooling of HDI processing prevented the special tool steel, Rex 121, from "over aging" and reacting with the Ni-P braze filler matrix while it might have transformed to martensite from the fast cooling after fusion. Microstructures of the Rex 121 containing suspension, is shown in Figure

34. The structure is dense and well adhered with a bi-modal high concentration of cubic phases, believed to be complex carbides. This bi-modal structure is very typical of highly wear resistant coatings where the larger carbides provide erosive wear resistance and the smaller carbides harden the matrix and prevents the matrix from eroding and leaving the larger carbides subject to pull out.

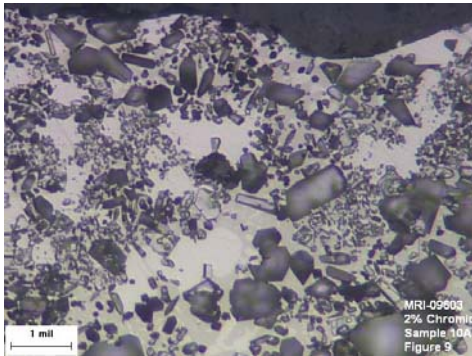


Figure 34. Metallographic Cross section of Sample #10 – TN 745Cr+Rex121 Vacuum Furnace fused.

The wear coupons in Figure 35 shows the macro-view of vacuum fused selected SRW wear scars. The figures are reproduced in the report differently to show the equivalent sizes (scale).

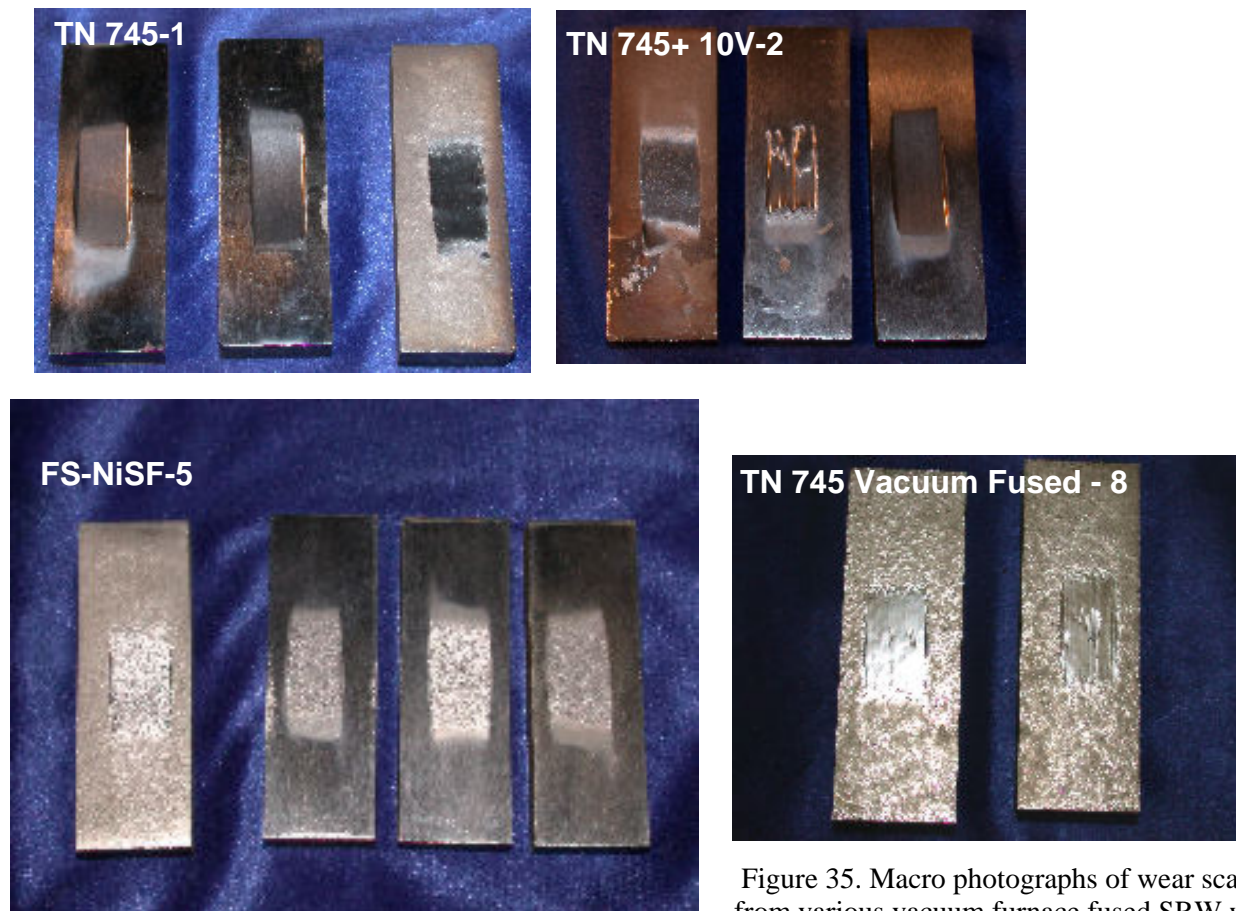


Figure 35. Macro photographs of wear scars from various vacuum furnace fused SRW wear test coupons.



Figure 35 (cont). Macro photographs of wear scars from various vacuum furnace fused coupons

Figures 36 through Figure 41 are metallographic cross sections of the various coatings showing how they performed in the SRW wear test

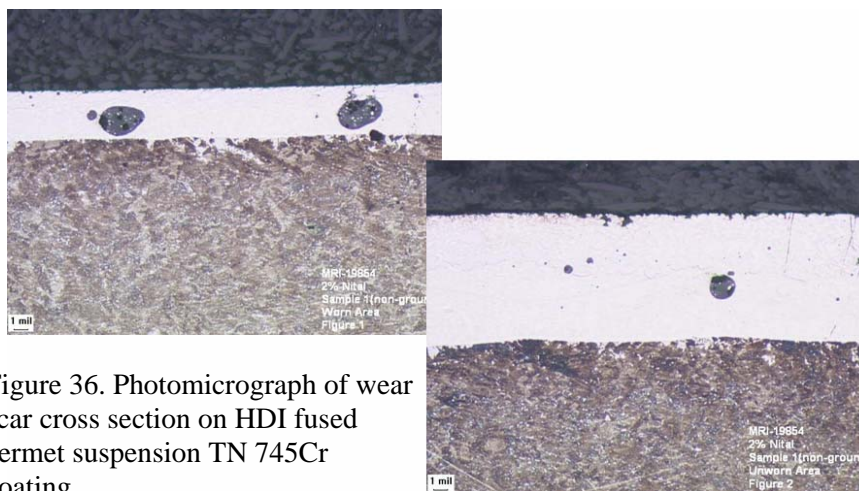


Figure 36. Photomicrograph of wear scar cross section on HDI fused cermet suspension TN 745Cr coating.

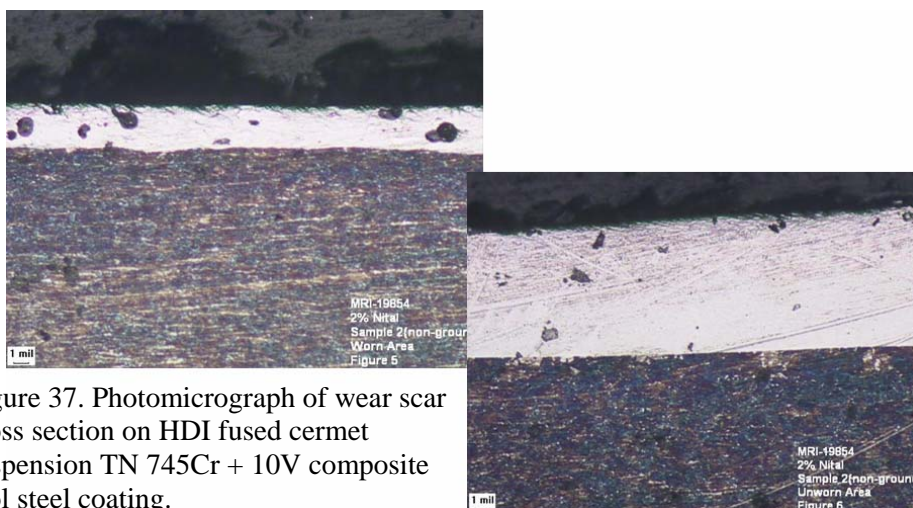


Figure 37. Photomicrograph of wear scar cross section on HDI fused cermet suspension TN 745Cr + 10V composite tool steel coating.

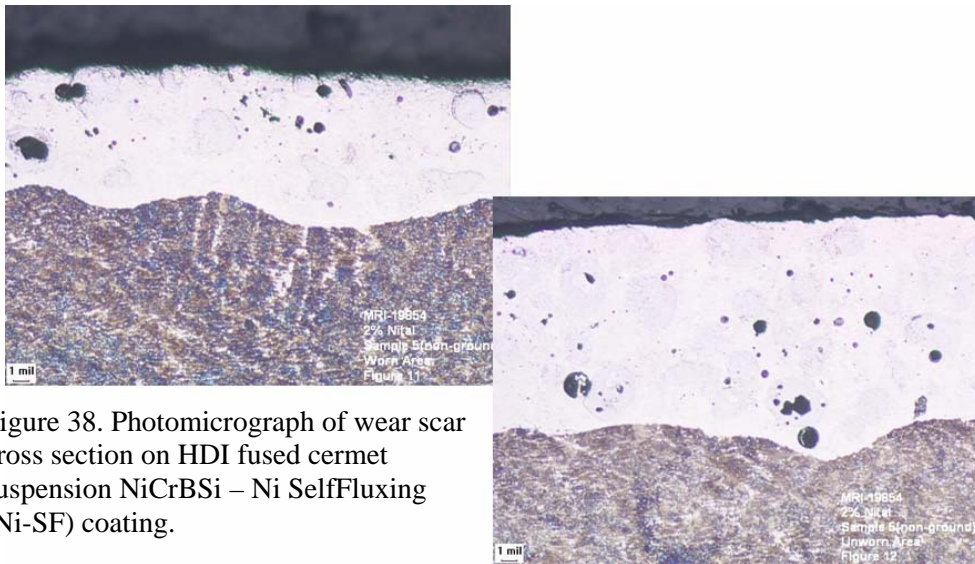


Figure 38. Photomicrograph of wear scar cross section on HDI fused cermet suspension NiCrBSi – Ni SelfFluxing (Ni-SF) coating.

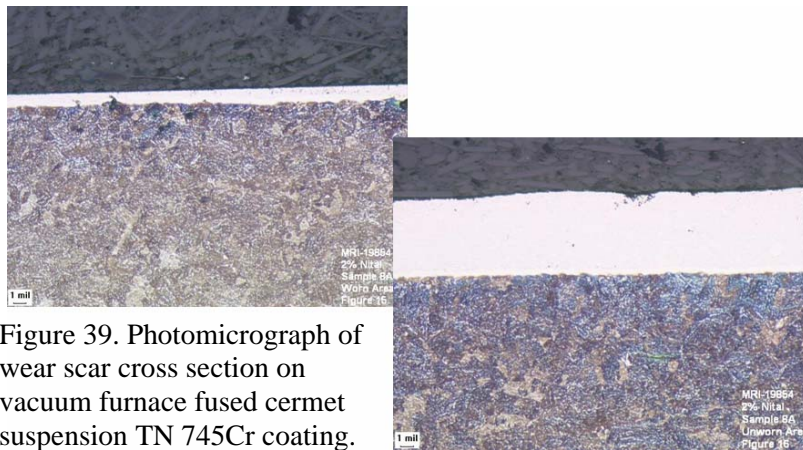


Figure 39. Photomicrograph of wear scar cross section on vacuum furnace fused cermet suspension TN 745Cr coating.

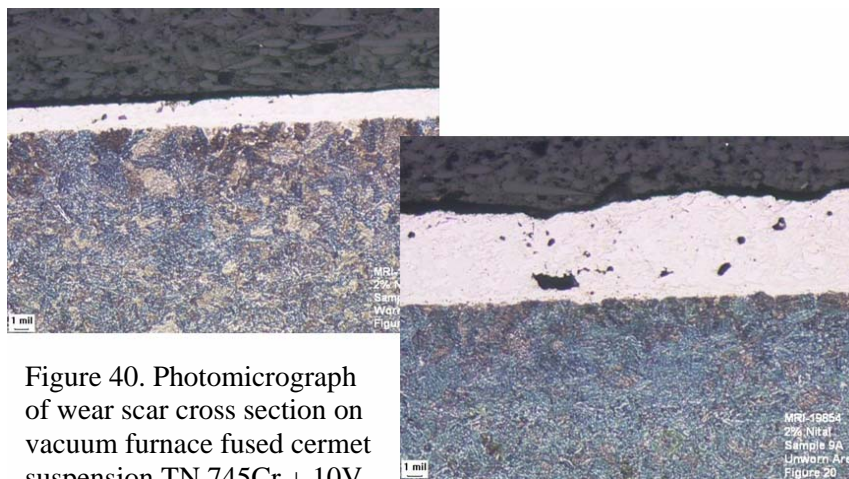


Figure 40. Photomicrograph of wear scar cross section on vacuum furnace fused cermet suspension TN 745Cr + 10V composite tool steel coating.



Figure 41. Photomicrograph of wear scar cross section on vacuum furnace fused cermet BrazeCoat Cr_3C_2 coating.

In summary, for SRW testing, the HDI fused suspension coatings and the HDI processed flame sprayed self fluxing NiCrBSi alloys and the vacuum fused coatings had nearly the same low wear rates with the vacuum fusing with being ~ 10% more resistant to sand rubber wheel abrasion. The exception, where an HDI fused coating was significantly better, was the suspension coating. This coating was a composite suspension coating with Rex 121 (tool steel) powder particles. Overall, it has been shown that the HDI process can be an excellent substitute for vacuum furnace processing. This is a central finding of this project, since the premise was that HDI fusion could be used to replace furnace fusing, especially in cases where the components are too large to be effectively furnace fused, either due to size or to a base materials thermal sensitivity. These results indicate that HDI fusion could in the required cases, replace vacuum furnace fusion and thus realize significant energy savings. At this point, it is nearly impossible to determine the scale of savings, since the industry and the applications are so fractionalized.

Table 1 also shows three “standards” to compare the HDI fused coatings against. These standard coatings were BrazeCoat® mats that were produced by vacuum fusing. These mat coatings were 10 times thicker and had 15% more carbide content. The HDI fused coating wear rates did very well against these “standard” high wear resistant coatings.

Figure 42 shows micrographs of the TN745Cr + Rex 121 suspension coatings that have been HDI and furnace fused. The structures are seen to be a complex mixture of phases with darker ‘carbide’ phases in a white matrix. This figure shows that the HDI process produces acicular (needle like) phases while the furnace fused suspension coating shows larger, more rounded dark phases. This phase difference is likely due to the much longer time at the liquidus temperature of the suspensions compared to the very short HDI fusion time (2-3 seconds vs. 3600 secs.).

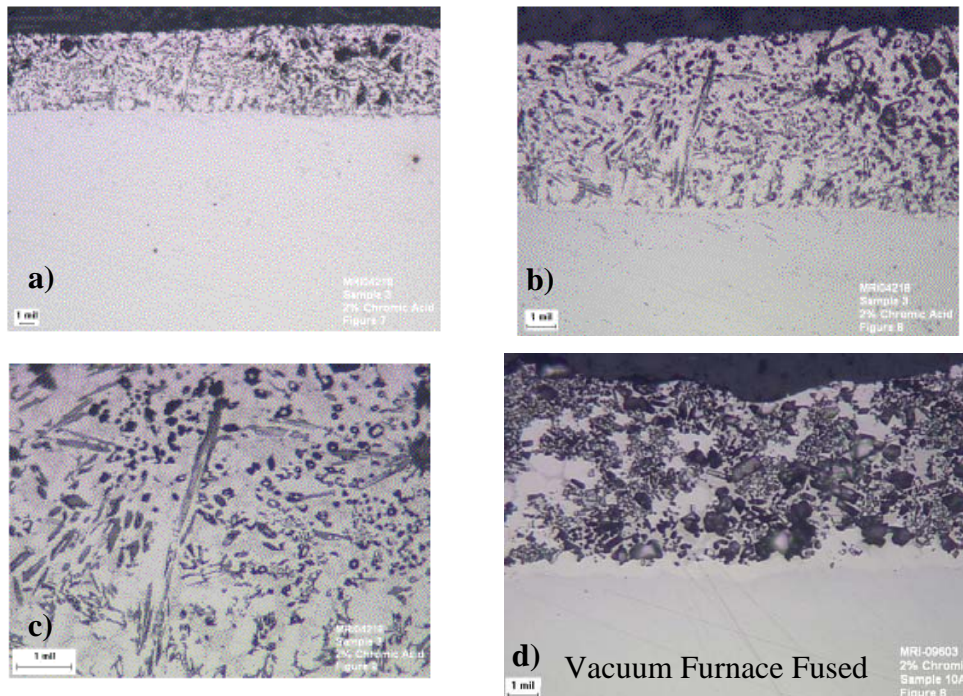


Figure 42. Metallographic cross section of TN 745Cr + Rex 121 coating a) suspension @ 100x, b) suspension @ 200x, c) suspension @ 400x, d) furnace fused @ 100x.

4.4.2 Pin on Disk Wear Testing

Pin on Disk (POD) testing was conducted at Crucible Research. In this test, pins were cut via electro-discharge machined from the HDI fused coated 3" x 3" x 0.5" steel coupons. The coatings were then machined flat. In the abrasion test, the coated end of the pin was then abraded against "garnet" paper. Figure 43 shows the test apparatus and set up. A summary abrasive wear results are shown in Table 2.

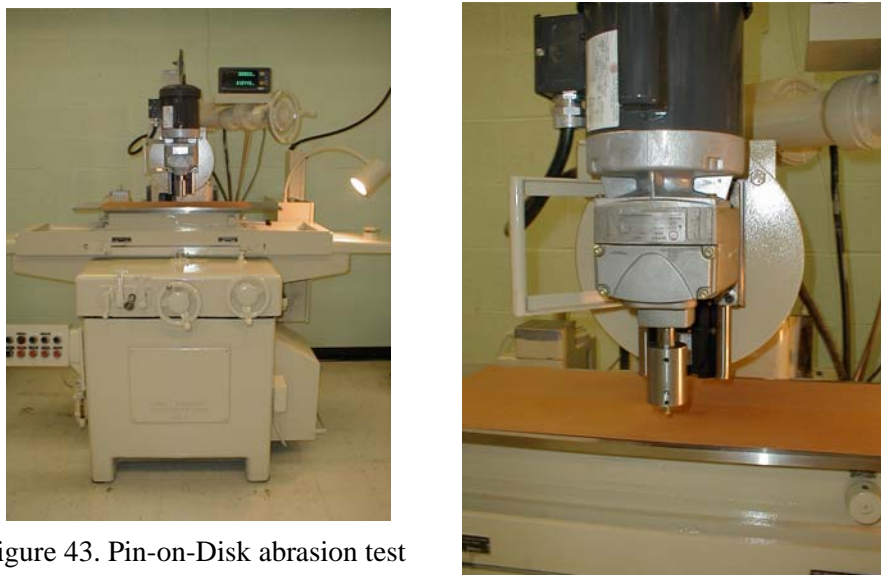


Figure 43. Pin-on-Disk abrasion test set up at Crucible Research.

Table 2. Pin-on-Disk Abrasion Tests for HDI and Furnace Fused (mg. loss of specimens)

	1	2	3	4	5	8	9	10	11
	HDI Fused 745	HDI Fused 745+10V	HDI Fused 745+Rex121	Tunnel Fused FSNi-SF	FSNi-SF (HDI)	Vac Fused 745	Vac Fused 745+10V	Vac Fused 745+ Rex121	Vac Fused BrazeCoat C
Test	26.5	54	13.4	18	27.2	123	37.7	27.4	11
	CPM M4	CPM 10V	CPM Rex 121						
Std.	50	32	2.5						

The POD tests and their relative wear rankings are very similar to the SRW testing shown in Table 1. The HDI fused TN745 + Rex 121 was still the best coating under these abrasion wear conditions. It should be noted however, that the vacuum fused results varied from the vacuum furnace fused wear rates being higher for the TN745 and lower for the TN745 + 10V. This inconsistency may be due to coating thickness variations, attributed to the fact that all POD coatings were ground before test and may have thinned the coatings too much. Comparisons to the standard Crucible P/M materials show the coatings, except for the TN745Cr + CPM10V, to be better than the base materials with the exception of the Rex 121 base materials.

Figure 44 is a chart of the comparative wear of the various HDI or vacuum furnace fused coatings. One can see the pin on disk abrasion (POD) and the sand rubber wheel abrasion (SRW) ranked the coatings similarly and that the HDI coatings, in most cases, had lower wear rates than the vacuum furnace fused coatings.

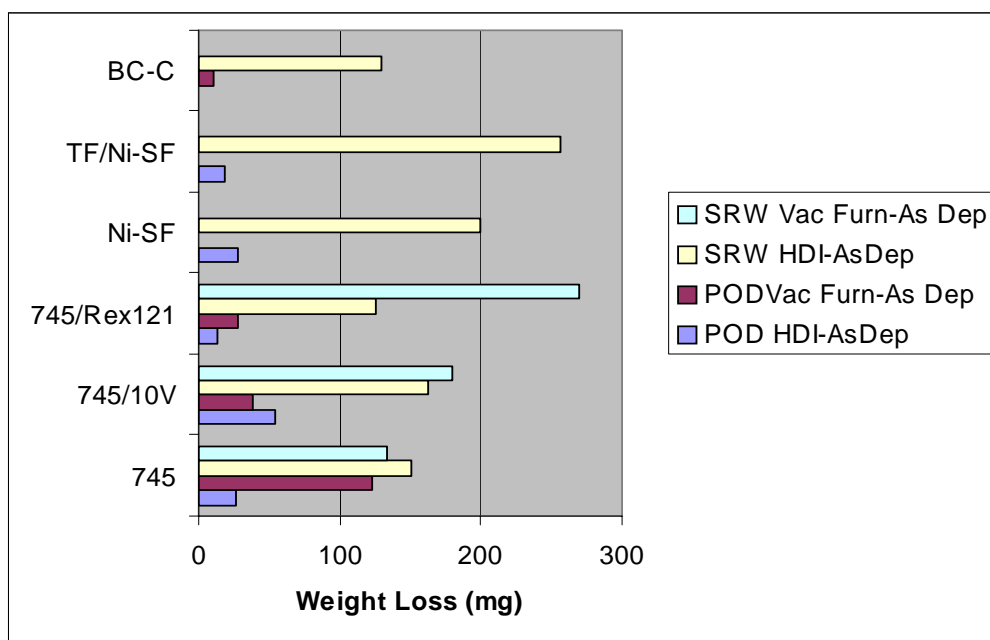


Figure 44. Comparison chart of Pin-on-Disk to Sand Rubber Wheel abrasion test results.

Figures 45 through 47 are additional photomicrographs that show the structure of the TN745, TN745+Rex 121 and the flame sprayed Ni-SF coatings. The coatings structures vary depending on the alloy composition.

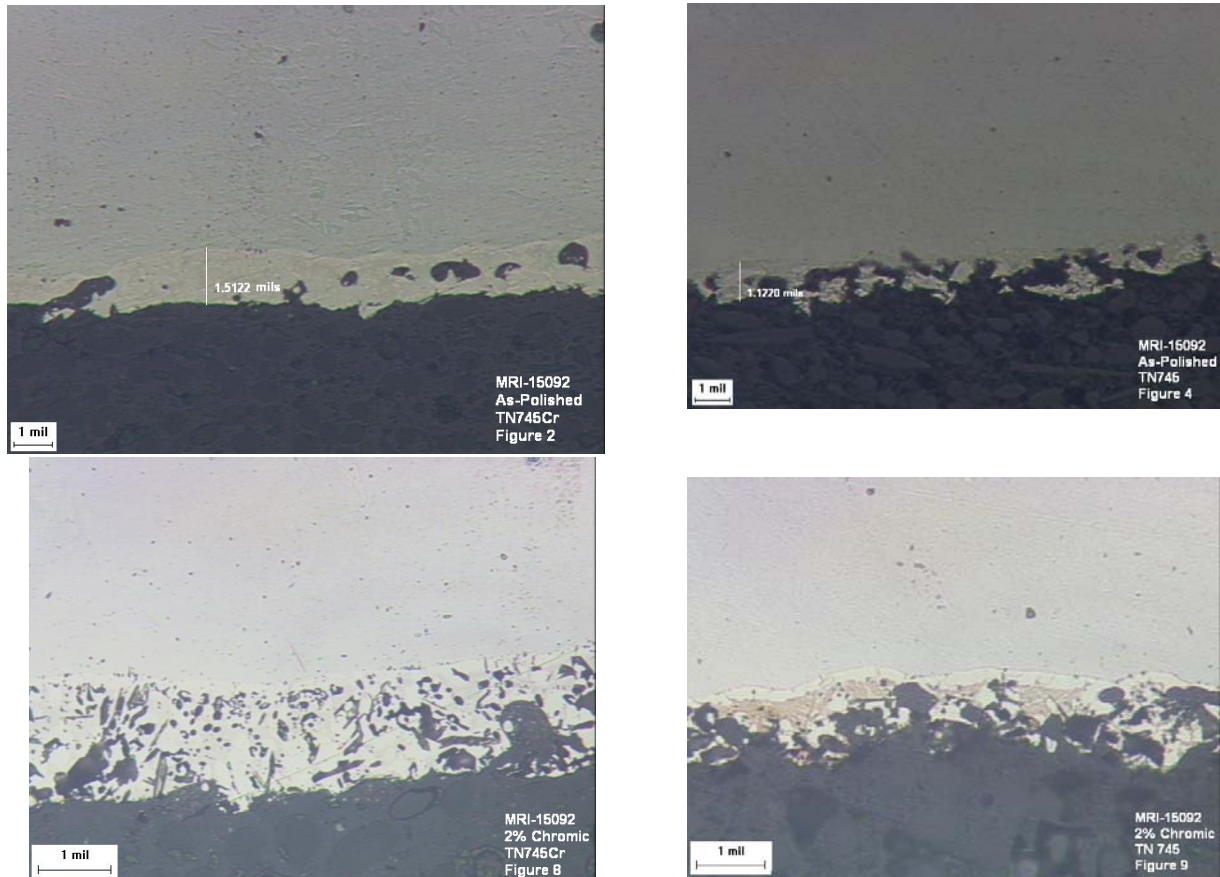


Figure 45. Micrograph of ORNL / HDI fused suspension coatings TN745 Cr ($\text{NiCrP-Cr}_3\text{C}_2$), coatings are etched.

Figure 45 illustrates that the pure braze filler (NiCrP) / carbide coating was the thinnest of the fused coatings processed at ORNL. The coating is however, completely fused and there is indication of the carbide going into solution and re-precipitating as “acicular” complex carbide phases (grey). These carbide were very hard and thus provided good wear resistance. Some other hard phases can also be seen, as well as some excessive oxidation, see the black phases in the polished and the etched cross sections. The HDI heating created more oxidation of this coating material compare to the other two coating materials shown in Figures 46 and 47.

Figure 46 illustrates a composite tool steel (Carpenter Rex 121) mixed with the TN 745Cr ($\text{NiCrP} + \text{Cr}_3\text{C}_2$). These coatings are much thicker $\sim 0.008''$ and more representative of typical wear coatings. The as-polished cross sections show the coating to be dense, low oxidation, and well fused and adhered. The carbide structure in the microphotograph shows that the HDI process did retain some original carbides (larger grey and blocky) and dissolved and re-precipitated some of the carbides, showing now as an acicular grey phase. There is also a phase that grows from the interface, believed also to be high in carbon

and likely some of the refractory metals from the Rex 121 that has been re-melted and re-solidified and mixed with the NiCrP braze alloy matrix. Overall the structure, although dense is more heated and melted than the goal... it had been hoped that the braze alloy matrix would melt preferentially and then the Rex 121 and the Cr_3C_2 phases would be wetted and braze joined. This is not the case in these HDI fused suspension coatings. It is apparent that much of the coating materials melted and fused, creating a unique, quenched structure that in itself will have high hardness and good wear resistance.

Figure 47 shows the relatively dense and oxide free structure of the NiCrBSiC (Ni-SF) coating that was initially flame spray coated before HDI fusing. The HDI fused structure is clean, dense and well adhered. There are no suspended carbides in this coating. The hardness of this coating stems from precipitated carbides, silicides and borides. The coatings are well within the coating thickness range of 0.008" – 0.010".

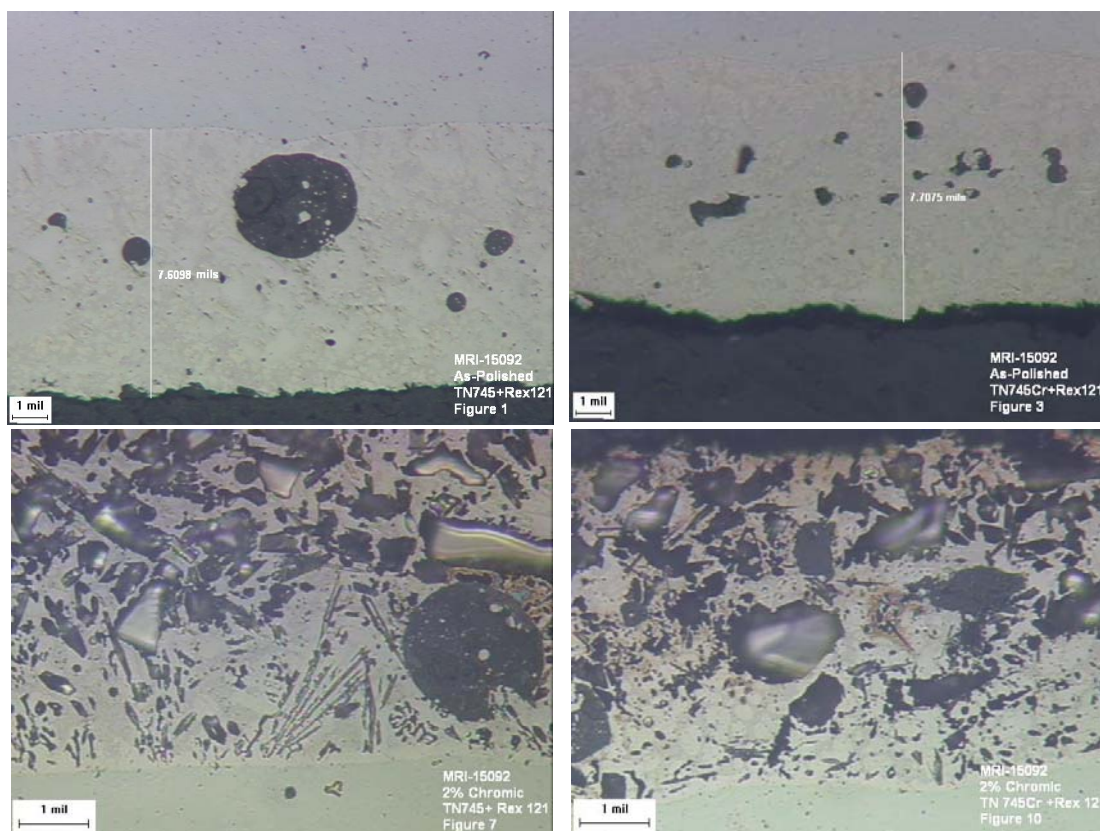


Figure 46 Micrograph of ORNL / HDI fused suspension coating: TN745Cr + Rex 121 (special tool steel) coatings, as polished (top) and etched (bottom).

Figures 45 through 47 showed the structure of the suspensions TN745 and TN 745 + Rex 121 and the flame sprayed Ni-SF HDI fused coatings. The overall objective of this round of work was to develop and produce coated specimens for sliding wear resistance. The TN745 and TN 745 + Rex 121 and the flame sprayed Ni-SF HDI fused) HDI coatings were selected for ASTM / sliding block on ring test.

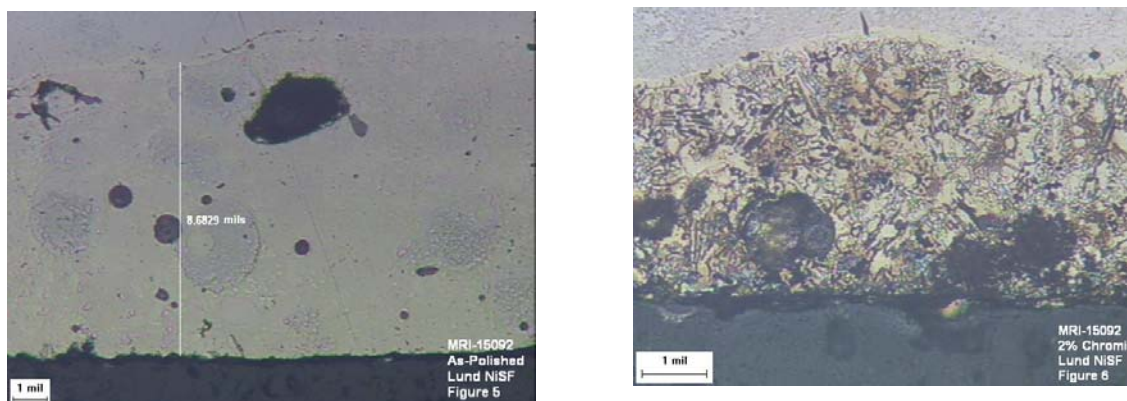


Figure 47. Microphotographs of ORNL / HDI fused flame sprayed NiCrBSi-SF coatings, as polished (R) and etched (L).

4.4.3 Sliding Wear Testing

In order to understand the sliding wear performance of selected HDI processed coatings, the ASTM G 77 test for Ranking Resistance of Materials to Sliding Wear was conducted at Falex on their block on ring test machine. In these tests, the blocks were the coated component and the rings were made from SAE 4620 steel hardened to R_c 58-60. The test load was 400 lbs and the ring speed was 200rpm and were run dry with no lubrication. The wear (mass loss) was measured after 2 hours and is reported in Table 3.

Table 3. ASTM G 77-Sliding Ring on Block Wear Test Results

	Wear		Coefficient of Friction
	Mass Loss (mg)		
Coating	Block	Ring	
HDI NiSF	0.0005	0.0238	0.128
	0.0026	0.0041	0.143
HDI Suspension TN745Cr-REX121	0	0.0593	0.134
	0.0006	0.0445	0.136

The results in Table 3 show that the HDI treated suspension composite tool steel-cermet coating (TN745Cr+ Rex 121) had almost no wear, indicating a very high wear resistance. The HDI fused Ni-SF (flame sprayed NiCrBSi) coating also had quite low wear. The friction coefficients were very similar between these coatings.

Figure 48 illustrates the sliding wear scars made by the rotating ring on the HDI fused TN 745Cr+Rex 121 composite-cermet suspension coating and the HDI fused flame sprayed Ni-SF coatings. These small scars, especially on the HDI processed suspension coatings indicate the potential for the HDI process for making highly wear resistant coatings.

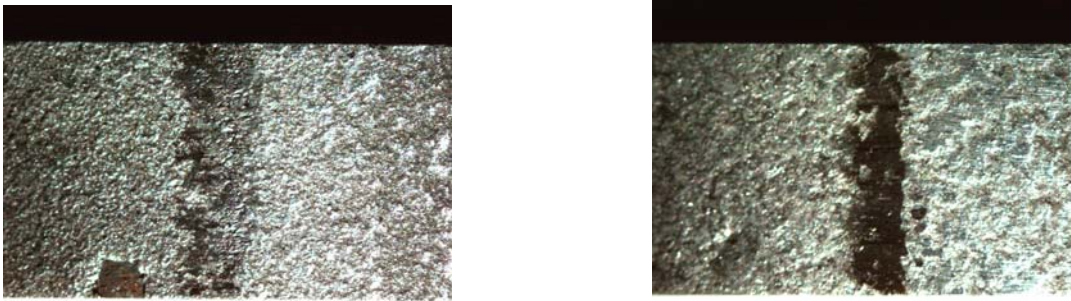


Figure 48. Photographs sliding wear scars on a) HDI fused TN 745Cr+Rex 121 composite-cermet suspension coating and b) the HDI fused flame sprayed Ni-SF coatings.

4.4.4 Summary of Processes & Wear Results

Vacuum Furnace Fusing

- Entire part needs to be heated and can be an issue with thermally sensitive materials
- Limits surface fusion to smaller parts
- More energy intensive than HDI
- Wear rates were low but comparable to HDI

HDI Fusing

- Only heats the surfaces of parts/ doesn't affect bulk materials.
- Enables larger parts to be coated
- More energy efficient than furnace fusing due to local heating and short cycles.
- Wear rates
 - o Most suspensions rates were similar to vacuum furnace fusing
 - o HDI fused TN74Cr + Rex 121 had lower wear rates than vacuum fused coatingIt was noted that something in the structure was more responsive to HDI processing
- Coatings are ~50% thinner than the vacuum furnace fusing but would work well on many parts.

4.4.5 Additional HDI Process Development

Figure 49 shows TN745Cr (NiCrP + Cr₃C₂) suspension coatings that were HDI fused by ORNL using their most optimum HDI processing, selected by their modeling. The process used a lower power scan, to preheat the base metal first and in the fusion scan the lamp accelerated as it neared the end of the coupons to prevent undue build up of heat. The coating surfaces in Figure 49 were uniform but with some "surface defects" resulting from the suspension binders out gassing near the edge of the specimens as the lamp traversed off the coupon.

The coating microstructures shown in Figure 50 are well fused TN 745 + Rex 121 composite suspension coating. The coating is dense, with isolated pores but was quite thin ~ 0.002". Work continued to the end of the project to get the coatings thicker. Figure 51 shows the increased surface hardness from the coating on the steel surface.

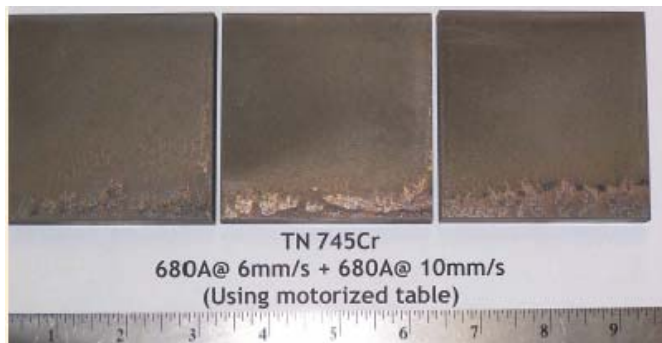


Figure 49. Pictures of ORNL / HDI TN745Cr (NiCrP + Cr_3C_2) fused suspension coatings.

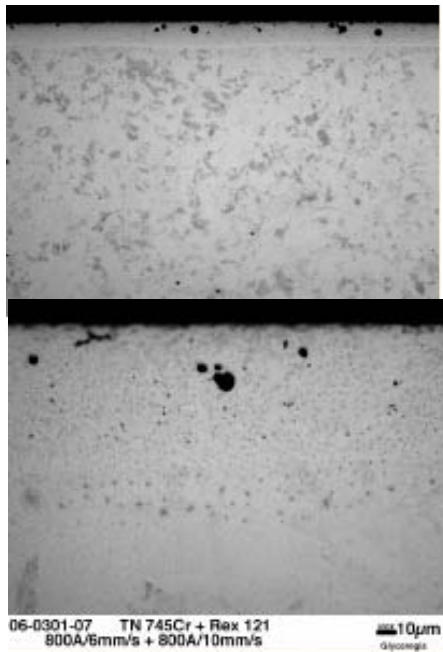


Figure 50. Micrograph of ORNL / HDI TN 745 + Rex 121 fused suspension coatings.

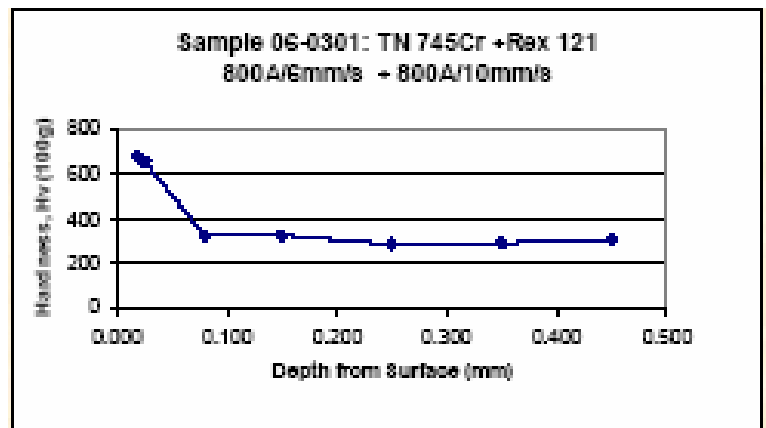


Figure 51. Microhardness trace of ORNL / HDI fused TN 745 + Rex 121 suspension coatings.

Figures 52 show the results achieved on the flame sprayed Ni-SF coatings. The coatings are dense, well fused but thin as well.

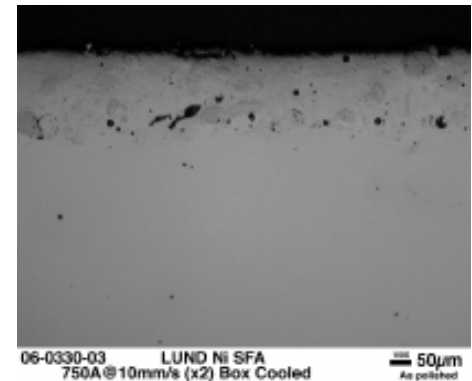


Figure 52. Micrograph of ORNL / HDI fused flame sprayed Ni-SF coatings.

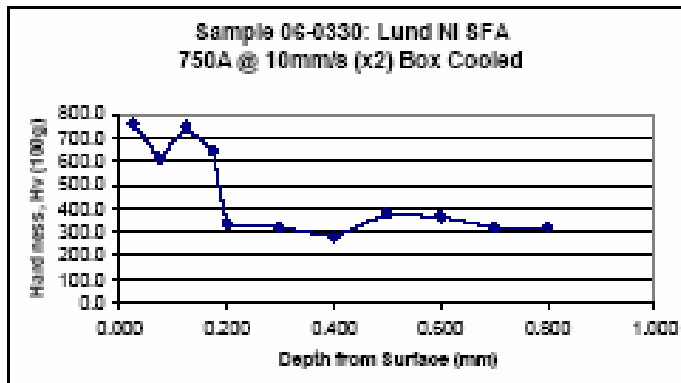


Figure 53. Microhardness trace of ORNL / HDI fused flame sprayed Ni-SF coatings.

Figures 51 and 53 show the hardness of the suspension TN 745 + Rex 121 and the flame sprayed Ni-SF HDI fused coatings. The hardness profiles show similar peak hardness values. But the Ni-SF coating was thicker and therefore harder to a deeper profile.

Overall the HDI coatings in this round of tests to make sliding wear coupons has been found to be different than when making similar coatings for the abrasion tests.

Pictures of laser fused suspension coatings are shown in Figure 54. These trials were initiated as a comparative evaluation of laser fusion to HDI. The big difference comparing HDI fused coatings to laser fused coating is that laser fusion only fuses a narrow pass at a time creating overlaps in the fusion structure as can be seen in Figure 54a and 55b. Note that HDI fused the entire 3" width in one pass over the coupon length. These laser fusion results show the coatings to be too fused and mixed with the base metals.

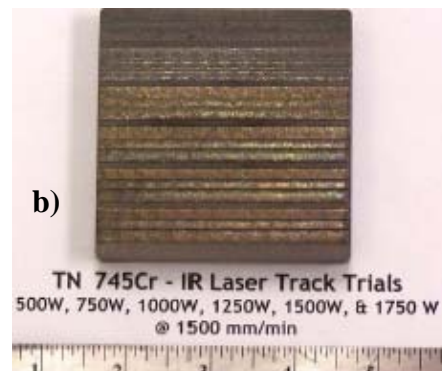
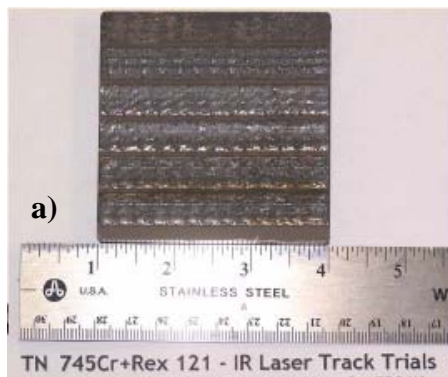


Figure 54. Pictures of ORNL / laser fused suspension coatings a) TN745_Rex 121 and b) TN745Cr

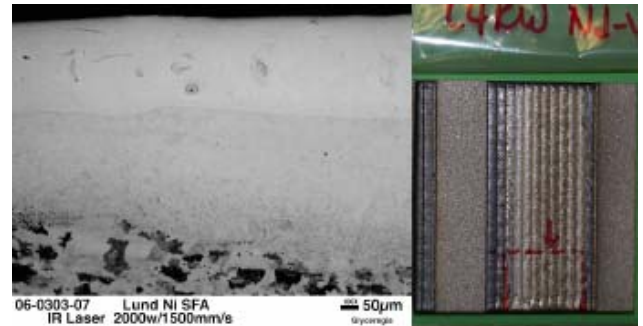
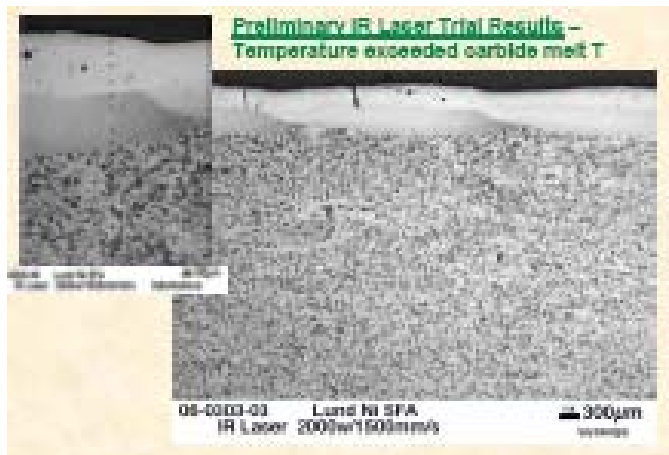


Figure 55. Microphotographs of ORNL / laser fused flame sprayed Ni-SF coatings.

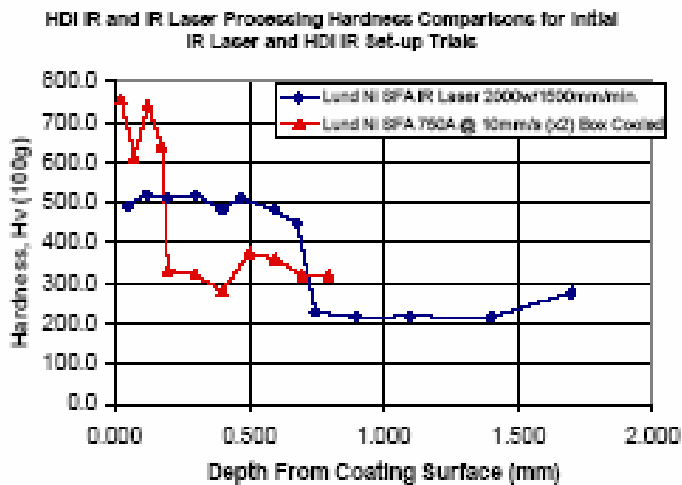


Figure 56. Microhardness trace of ORNL / laser fused flame sprayed Ni-SF coatings.

Note that in the laser heating there was considerable mixing with the base metal. This result can be seen in the hardness profile of the flame sprayed Ni-SF laser fused coating where the laser treated coating is shown to be softer at the outer surface, but harder deeper into the base material, where the base steel was melted and rapidly quenched.

4.5 Task 5.0 Field Testing

The aim of this task was to work with the industrial partners to develop applications that would benefit from the use of HDI processing, selecting components that were capable of being fused in the ORNL system while capable of testing the capabilities of the HDI coatings.

Initially the project had selected several applications;

1. Densification nickel claddings for cookwear (Ametek),
2. Fusion of corrosion resistant stainless steel thermal spray coatings on large components for use in chemical plants (Carpenter)
3. Wear coatings on large rolls used in metallurgical plants (MRi and Crucible) and

4. Agricultural equipment and blades (Lund International). Based in the first year down selections and the re-focus on fundamental studies the only remaining industrial partner with an application within the capabilities of the HDI unit at ORNL was a agricultural harvester cutting blade.

The agricultural blade application was of the appropriate size and geometry (flat) and had a good base of comparison. Therefore, it was decided when the project plan for Years 2-4 was defined, to exclusively focus on the blade application.

4.5.1 Lund Agricultural Cutting Blades

Lund International, a project partner contributed over 75 agricultural blades samples for testing both thermal spray and suspension precursor layers. Figure 56 shows the test set up that was eventually constructed at ORNL to fuse larger flat surfaces and was suitable of the fusion of Lund's flat ~ 4" long harvester blades. Lund's standard coatings for these blades were a flames spray Ni Self Fluxing (NiCrBSiC) coating that was subsequently tunnel/belt furnace fused then re-hardened. The project aimed at developing an HDI process that would fuse the flame sprayed Ni Self Fluxing coating on the actual blades, building on the developments from the HDI processed 3" x 3" steel coupons discussed in the previous sections of this report. Figure 57 illustrates the positioning of the IR Lamp, the construction and use of a water cooled protective atmosphere chamber with quartz window for the IR transmission

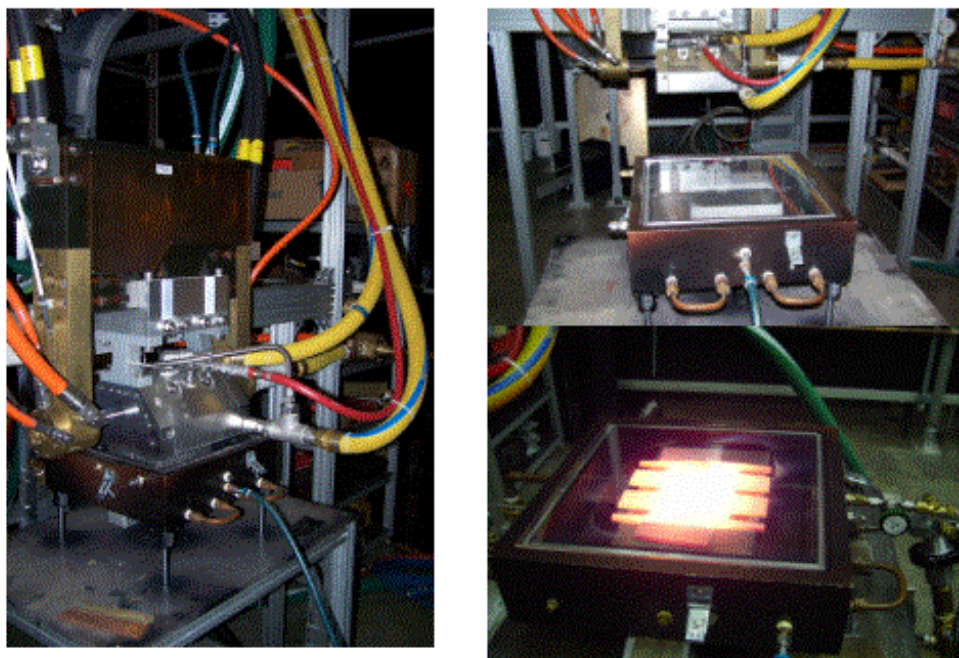


Figure 57. Pictures of the Lund Blade fusing set up at ORNL's HDI cell: a) the layout of the lamp and enclosure set-up and b) the fixturing of the blades in preparation for HDI fusion.

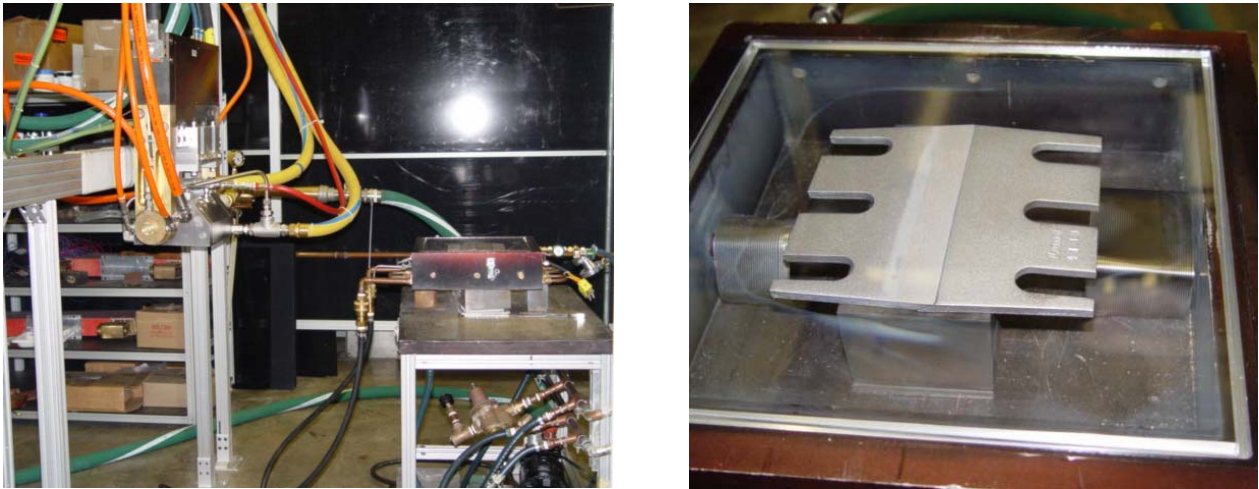


Figure 57 (cont). Pictures of the Lund Blade fusing set up at ORNL's HDI cell: a) the layout of the lamp and enclosure set-up and b) the fixturing of the blades in preparation for HDI fusion.

The blades were positioned in the atmosphere chamber as shown in Figure 57 in such a way that one blade cutting tapered edge was the heat sink for the other, creating a more uniform heating cross section. As in the coupon coating, the HDI process eventually consisted of a few preheating passes at lower power followed by a higher power, accelerating pass. Process development was conducted on 75 coated blanks. Metallography and hardness was completed on "as-received" materials. It was found that the precursor coating varied in thickness from ~0.008" - 0.020" thick coating. The coating surface hardness of the as-received varies (~600 - 850 H_v). A series of HDI processing trials were conducted on Lund blade trials utilizing the 750 kw HDI plasma arc lamp. This larger HDI lamp was chosen for this application since it is equipped with a uniform irradiance reflector that allows a uniform thermal heat flux and simultaneous processing of the 6 inch wide blade. Hence, this lamp will minimize the IR processing time. Figure 58 shows the coated and HDI processed blade edges after heating. Thermal processing trials were conducted and the results from these are reported below.

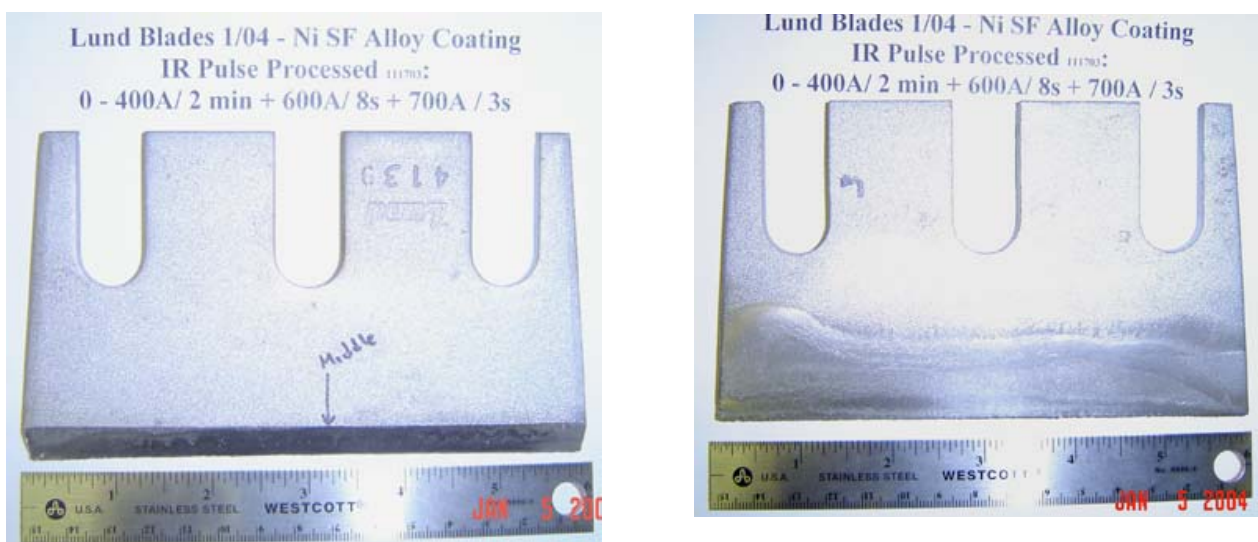


Figure 58. Pictures of the Lund Blade after fusing set up at ORNL's HDI cell.

The coating results from the 750 Kw, 500 Amp trials are shown in Figure 59. The porosity for the best trial appears to be (<5% est.). Extraordinary hardness values were achieved on the HDI processed, Nickel Self-Fluxing flame spray applied coatings. The carbide particles in the fused coating were found to be evenly distributed and returned to a spherical shape (indicating there was sufficient heat to liquefy the surrounding matrix and sufficient time to allow the carbide agglomerate to reform into an equilibrium state). Also, as desired, neither surface nor interface carbides exhibit melting, and the matrix interface appears “white”. The apparent cause of the observed porosity are most likely the result of entrapped gases (from flame spray) or oxidized surfaces (either due to “burn” in the flame or post-spraying infiltration of contaminants). Previous experience on other coatings suggest that further optimization of the HDI processing parameters would help eliminate/reduce some of this porosity. Some preheating and/or post-heating prior/after HDI processing may enable sufficient time to allow some of these entrapped gases to migrate to the surface and release. In addition, modeling developments achieved on flat samples have demonstrated that HDI processing parameters can be successfully predicted and developed for flat samples. So, HDI process parameter optimization seems quite achievable for these chevron-shaped edged blades as well. The hardness profile taken in these blades show that coating edge hardness increased significantly over the as coated hardness values of 600 – 850 H_v.

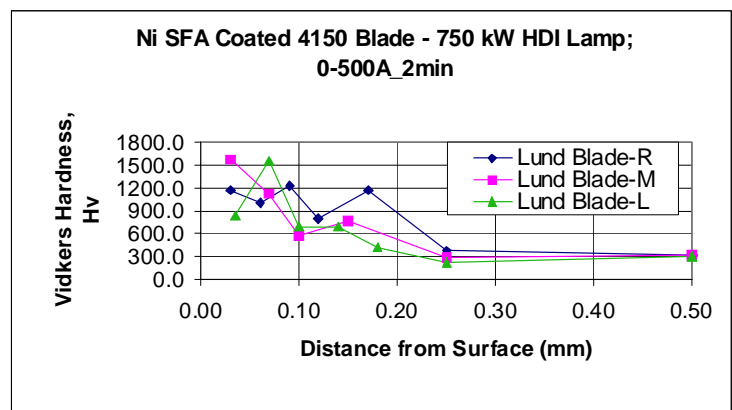
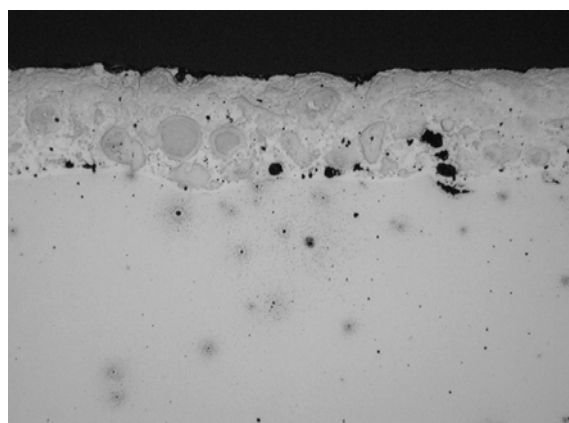
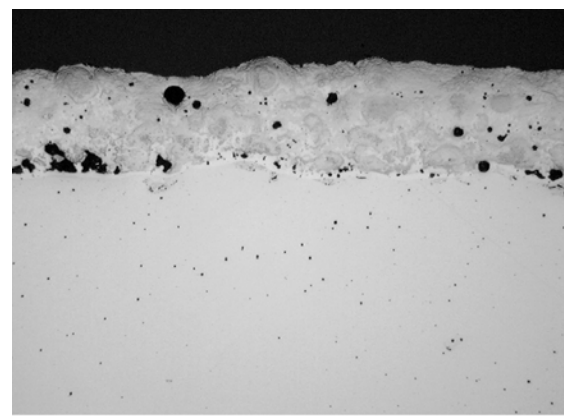
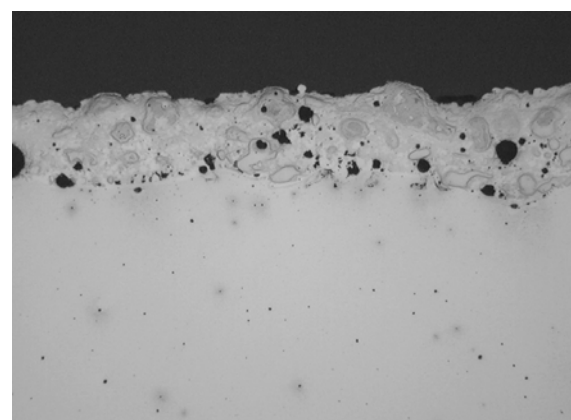


Figure 59. Lund Blade IR Processed: 0-500 A/ 2 min., showing coating structures and coating hardness profiles.

Figure 60, shows the additional hardness results of additional testing that was completed. Comparing the hardness profile of the HDI fused Flame Sprayed NiCr Self Fluxing coatings to the same type coatings fused conventionally by Lund International, it can be seen that the HDI fused blade coatings have much higher hardness values.

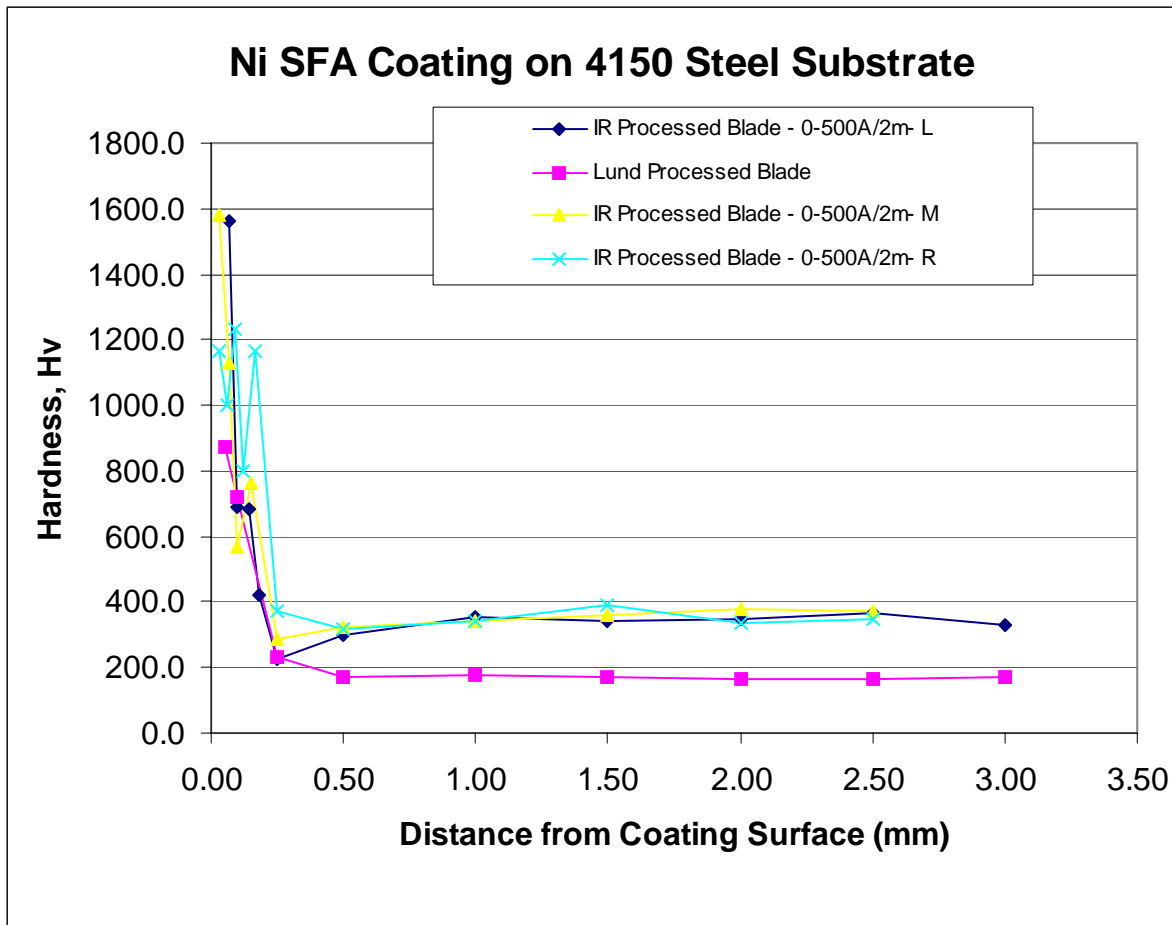


Figure 60. Lund Blade IR Processed (HDI fused) vs. Lund process coating hardness profiles.

Using these HDI process parameters, twenty five (25) cutting blades were HDI fused at ORNL for Lund International to place in field test. It was expected that results from field tests would have been completed by the end of the project, however personnel and facility changes at Lund kept these tests from being completed. The work in the overall task demonstrated the capability for HDI process to produce high quality fused coatings, although there are process improvements to be made, such as only edge heating of the blades and the scaling of the HDI process to fuse larger blades and shear cutting bars.

Additional HDI Testing with Industrial Application

Another application area for HDI fusing was for placing hard, fused coatings on thermally sensitive materials, for example aluminum. Wear coatings on aluminum have long been sought after for use in

automotive applications such as engine cylinders as well as for use in other transportation vehicles where sliding wear and surface galling is a problems. HDI applied carbide coatings may offer some advantage.

In this application, aluminum melts well below the actual fusion temperature of the applied suspension/composite wear coatings, 650 vs. 950°C. The project did complete some scoping studies of applied suspension precursors (TN8020 Cr with Cr_3C_2 and TN8020W that contained WC particles). In these trials a CuNiMn braze filler was used in place of the NiP or NiCrP matrices. The CuNiMn was more compatible when fused and mixed with the aluminum base metals.

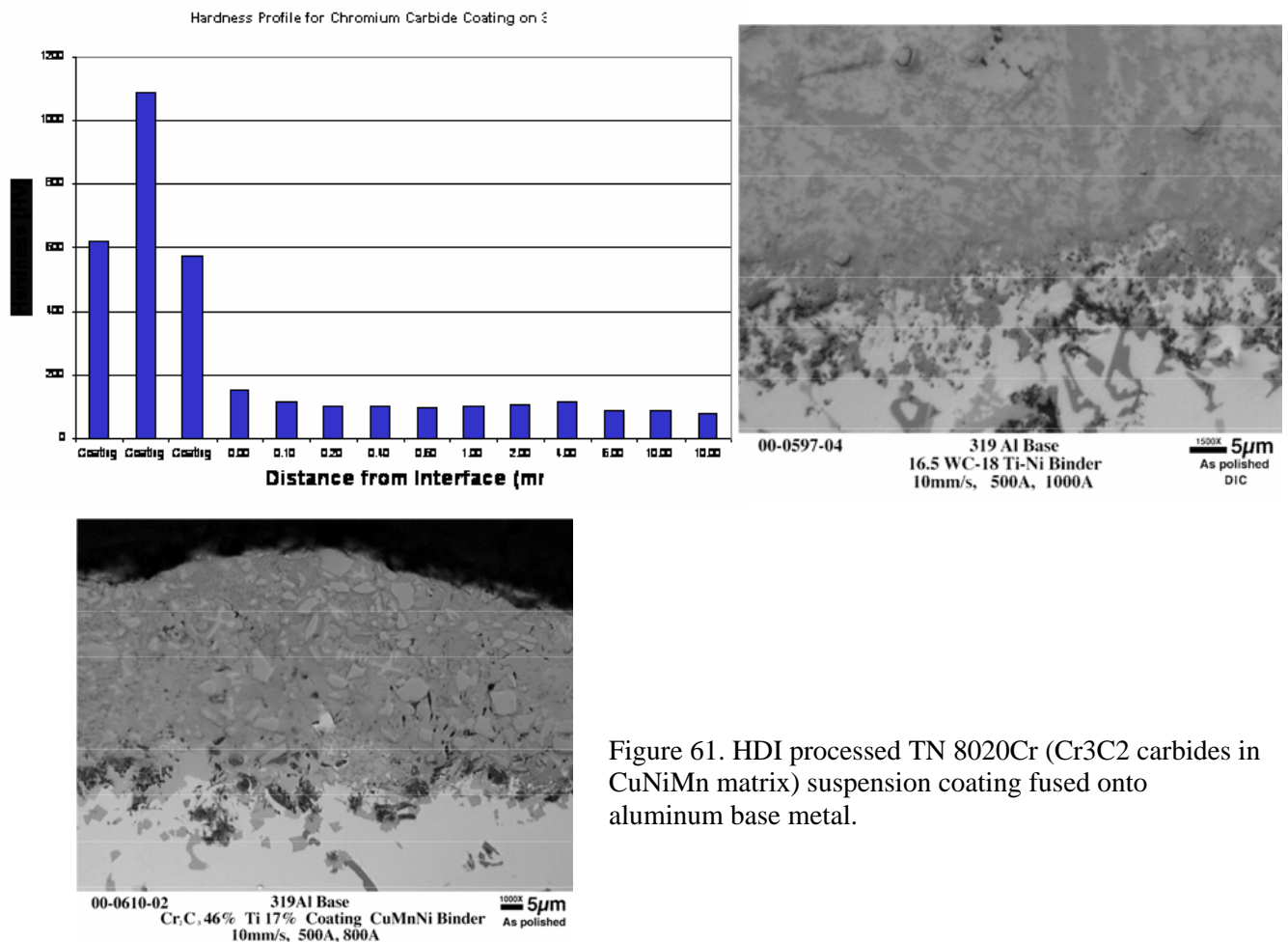


Figure 61. HDI processed TN 8020Cr (Cr_3C_2 carbides in CuNiMn matrix) suspension coating fused onto aluminum base metal.

Note the significant surface hardening of the coating, with hardness values from 800 – 1100 H_v , compared to the base aluminum hardness of ~ 100 H_v . The Cr_3C_2 particles and matrix in Figure 60, show them to be well fused, adhered and dense with a base metal interaction zone that was less than 5 microns. The metallurgical interaction zone does not have an apparent brittle intermetallic zone, a major problem with many fused coatings on aluminum.

Figure 62 illustrates a similar HDI fused wear coatings with WC particles. The coatings are thin, but as in the case of the Cr_3C_2 reinforced coatings, seen in Figure 61, these coating are hard, dense and well adhered.

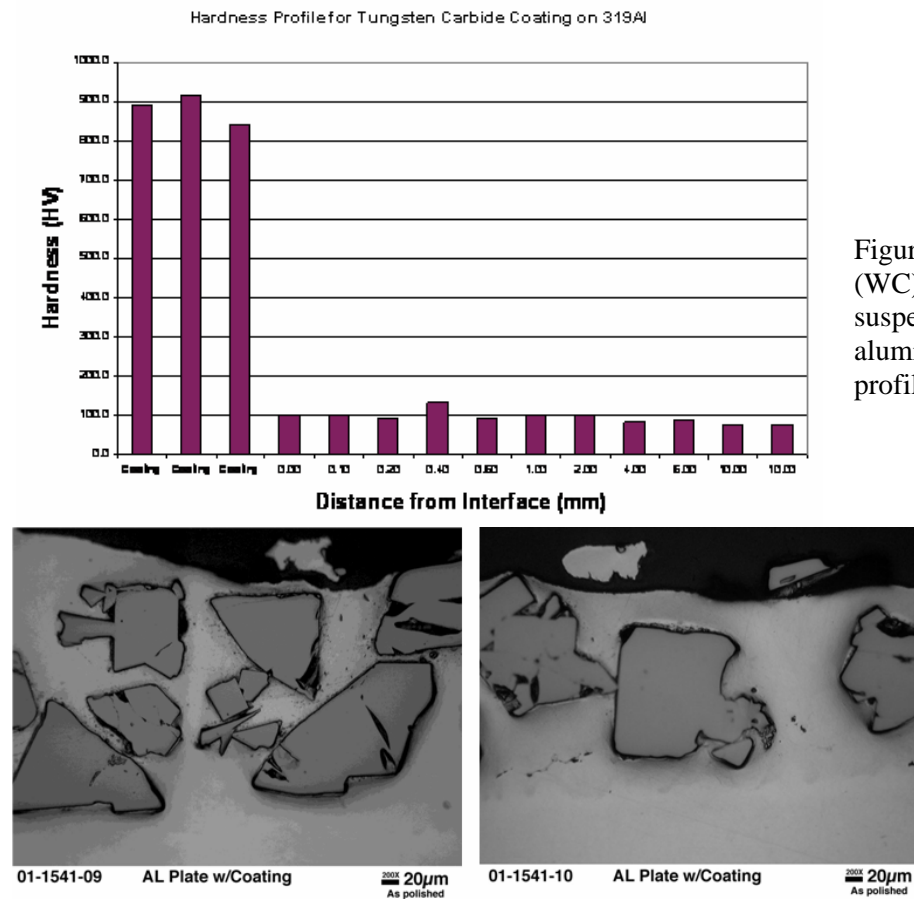


Figure 62. HDI processed TN 8020 W (WC) particles in a CuNiMn matrix, suspension coating fused onto aluminum base metal; a) hardness profile and b) coating structure.

This work showed the potential for placing wear coatings on aluminum base materials. Due to the need to focus on a set of materials and applications, this coating development was not advanced beyond this feasibility demonstration. The project concluded before this industrial application area was supported. However, the feasibility was demonstrated and likely has high potential in commercial vehicles and in industrial components where aluminum would be preferable over steel if economic sliding wear and gall resistant coatings were available.

5.0 ACCOMPLISHMENTS

The project accomplished many of its technical goals but fell short on developing a specific commercial application. Over all the following goals were achieved.

- An understanding of the HDI coating fusion process was developed.
- An operational computational process model was developed and its effectiveness was demonstrated
- A set of precursor materials for producing wear coatings were identified, studied and the wear properties were measured.
- An HDI process was developed for fusing flame sprayed Ni self fluxing alloys and carbide (cermet) suspension applied coatings.
- A new composite carbide-tool steel suspension coating was developed and demonstrated to be better than vacuum furnace fused carbide coatings.
- The HDI process was demonstrated to be able to fuse flame spray coatings onto 4" agricultural blades and yield improved coating hardness.

6.0 CONCLUSIONS

The project evaluated and developed an understanding of how high density infrared heating technology could be used to improve infiltrated carbide wear coatings and/or to densify sprayed coatings. High Density Infrared (HDI) heating systems and techniques were demonstrated to be able to fuse thermally sprayed coating and to infiltrate carbide coatings for use in applications such as agricultural blades, rolls for metallurgical processing, construction and mining vehicles, components for paper and polymer processing as well as for a range of aerospace applications. Engineering development focused on developing process and equipment technology and know-how to implement HDI systems that could fuse coatings on a range and size of components. Fundamental research and process modeling was conducted that developed some understanding of the interaction of HDI processing with the coating materials and the base materials, and determined selected coating properties, focusing in particular on wear resistance.

The research included HDI fusion evaluations of infiltrated carbide suspensions such (BrazeCoat® S), composite suspensions with tool steel powders, thermally sprayed Ni-Cr- B-Si (self fluxing alloy) and nickel powder layers. The applied work developed practical HDI / transient liquid coating (TLC) procedures on test plates that demonstrated the ability to fuse carbide coatings for industrial applications such as agricultural blades, construction and mining vehicles. Fundamental studies helped create process models that led to improved process understanding and control.

The following conclusions can be made:

- HDI coating fusion can produce excellent wear resistant coatings.
- HDI was effective surface fusing heat source.
- The coating HDI fusion process, with proper precursor conditions, was rapid and could be focused on specific areas.
- A proper degree of preheat in the base materials was required.
- Preheat could be done using lower power irradiation passes before the high fusing power passes.
- The "brazing type" suspension composite carbide and tool steel and the flame sprayed NiCrBSi (self fluxing) coating responded best to HDI processing.
- HDI fusing was found to be an improvement over flame fusing
- HDI fused coatings had better wear and less base effects than flame and furnace fused coatings.

- The wear rates on coatings by both HDI fused NiSF and the suspension coatings were near equivalent or lower than vacuum furnace fusing while the HDI coatings were~ 50% thinner.
- HDI fusing would offer energy savings over heating the entire component to fusing temperature.
- HDI fusion was shown to a promising fusion method compatible with wide range of coating and base metals.
- The HDI process was found to be very sensitive to coating precursors and compositions
- Precursor layers have to be compatible with IR heating and with wetting and reactions with the base materials.
- The precursor layers had to be sufficiently thin, dense and oxide free to be able to effectively transfer the high intensity surface heating.
- An HDI process model proved to be an effective process design tool.
- The ORNL developed thermal model that was developed facilitated the selection of HDI process parameter
- More effective heating schemes, including the use of preheat passes to smooth out thermal gradient and the use of accelerating passes as edges were approached, preventing overheating.
- It was found that for each application and material set, specific developments were needed to tailor HDI process to actual parts.
- The ORNL HDI process model proved to be effective in shortening the development cycle for parameters, especially for the suspension and the flame spray coated wear test samples.
- A shortcoming of the project was the inability to achieve repeatable, uniform coating structures and thicknesses. In this work, after settling on a given set of parameters, subsequent repetition of the coating for the wear test samples led to variations that were not understood. The variations may have been the precursor or HDI set up variables. With the conclusion of the project, a study of the repeatability and a systemic study of the influence of the coating process variables on coating structure and properties were not completed.

The industrial/commercial coating development centered on wear coatings on agricultural cutting blades that utilized flame sprayed NiCrBSi (Ni-self fluxing) coatings. ORNL with cooperation of Lund International developed a successful coating process that yielded 25 harvester blades for field testing. These blades at the conclusion of the program, had not found their way to field tests.

7.0 RECOMMENDATIONS

In summary the following recommendations are made:

- Further study the improvements in wear resistance that the HDI fused cermet-tool steel suspension coatings demonstrated: This recommendation stems from the result of the extreme wear resistance measured in a thin $< 0.005''$ thick coating. These coatings consisted of Crucible Research's Rex 121 powders added to the carbide-braze alloy composition that when rapidly quenched, consistently produced superior wear resistance. MRi and Crucible should work together to pursue the commercial application of the combination of their tool steel powders and the cermet suspensions and verify the performance improvements that HDI fusion processing can provide.
- Develop the HDI process for coating larger components and cylindrical surfaces: the HDI processing facility at ORNL could only fuse up to 12'' x 12'' flat surfaces. The real advantage of the HDI process will be its ability to fuse large components that would be either too expensive or too large to place in furnaces for fusion. Many of the wear components that would benefit from the developed wear coatings would have larger cylindrical surfaces such as rolls for paper or metals processing.
- Improve the HDI coating fusion process to be able to reliably and repeatably coat surfaces: Investigations showed that excellent HDI fused coatings could be applied, however, it was also found that significant process variations occurred, with many significant variables stemming from the pre-cursor layers.
- Seek applications in paper mills, polymer processing, metalworking and glass industries for coating of rolls and molds. Due to various project factors, the industrial application was narrowed to the agricultural blade. It suited the 12'' x 12'' flat surface fusing limitation of the ORNL HDI system and met a need of an industrial partner, Lund International. However, the real success would be coating larger rolls with wear coatings since such large rolls cannot generally be furnace fused.

The HDI process was demonstrated to be an effective fusing process for certain suspension and thermally sprayed coatings. Excellent abrasive and sliding "carbide-tool steel" composite suspension wear coating properties were produced; however the repeatability of the process on making carbide reinforced coatings, in the end, was not demonstrated. Additional process development is recommended based on the excellent, but variable wear results. Due to time and funding constraints, evaluations of HDI densification of thermally sprayed corrosion and wear coatings, although promising, were not able to be completed.

The HDI process did show promise in its ability to densify thermally sprayed carbide particle reinforced coatings and tool steel. HDI was shown to be a very broad, intense heating process that can surface melt layers, but the properties of the pre-HDI precursor layers had a large influence on the HDI processing parameters. Suspension coatings had a binder and low thermal conductivity while thermally sprayed layers had much higher densities but normally were much thicker. The HDI power and traverse rate needed to be adjusted and variations in the underlying structures led to coating densification differences. A limitation is that the HDI process robustness is dependent on the underlying materials and their structures. It was also found that wetting of underlying layers was affected locally by chemistry and that surface molten metal wetting elements such as B, Si and P were beneficial in achieving adherent HDI processed coatings. More work would be required to better understand these effects, especially on coatings that would have. If metal layers were already intimately bonded, such as the thermal spray

coatings made via High Velocity Oxy-Fuel (HVOF), then coating adherence was not found to be as factor. The HDI process was indeed capable of fusion and in the case of thin carbide reinforced suspension applied coatings, excellent coatings were achieved. However; due to the binder and poor thermal conductivity of the suspensions precursors, coating thicknesses much over 0.2 mm (0.005") were not able to be densified. Improvements in understanding the "debinding" steps in HDI processing needs to be developed.

It is also recommended that the HDI process variability be better understood focusing on the carbide-tool steel composite suspension precursors systems. Based on wear testing, these materials showed the most promise as an industrial coating, provided the HDI process can be scaled to larger plates and cylindrical shapes.

With equipment capability constraints, only fused flat surfaces ~ 12" on length were evaluated. In the wide range of applications that are possible, larger flat and large cylindrical surfaces would have to be coated. The work in the project centered on flat surfaces that could be densified in one pass. What would be needed is to modify the HDI equipment to heat and fuse coatings on larger flat (up to 36") and cylindrical surfaces (up to 24" diameter to start) and studying the interaction of the coatings with multiple, overlapping pass fusion.

Finally, it is recommended that after the appropriate HDI process scaling is established, that additional industrial applications be sought in the following areas:

Agriculture:	blades – (cutting corn, harvesting, etc.), equipment for bio-mass gasification systems
Glass:	wear resistant coatings on dies and transfer line components
Petroleum:	pump components (corrosion)
Metal:	steel processing and transfer rolls steel – hot rolling and cold rolling
Energy:	boilers , corrosion-resistant coatings
Transportation:	automotive/truck; engine and power transmission,
Mining:	earthmoving, material transfer systems

APPENDIX 1

Measured Thermal Properties of Thermal Spray and Suspension Materials

MATERIAL	CTE	Density (g/cm ³)	Thermal Conductivity [W/m*K]	Thermal Diffusivity [cm ² /s]	Heat Capacity [J/(g*K)]	Liquidus T [°C]
TN 660W	NA	6.27		100 - 500C: 0.018 600 - 800C: 0.024		
TN700	NA	4.60		100 - 500C: 0.003 600 - 800C: 0.005	150 - 500C: 0.49-0.59 600C - 800C: 0.0.59	
TN745Cr	NA	4.36		100 - 500C: 0.005 600 - 800C: 0.006		
TN745Cr + REX121+TN7 00 + binder	NA	4.68	100 - 500C: 0.9-1.0 600 - 800C: 0.9 - 1.5	100 - 500C: 0.006 600 - 800C: 0.008	100 - 800C: 0.56-0.57	
TN745Cr +10V +TN700 + binder	NA	4.24		100 - 500C: 0.004 600 - 800C: 0.005		
Ni SFA (Metco 16C)	20 - 600C: 11- 12.7 600 - 900C: 12.7-14.3	7.74	150-500C: 18-23 600-800C: 62-69	150 - 500C: 0.046 600C - 800C: 0.055	150 - 500C: 0.52-0.61 600C - 800C: 0.63-0.70	
4340 (Ni-Cr-Mo)	3.5mm/mm degC	7.83	37-42			15044 - 1520 (=TMP)
1008/1010/10 20	10.5 @675 oC	~7.86	RT: 11.3 [65.3] 600 oC : 63.0 [36.4] 800 oC: 49.3 [28.5] [Btu in* ft- 2*h-1 * oF]			1516
316L	20 - 400C: 15-18	7.95	16+			1405-1445 (TMP)

	600 – 800C: 19					
90V hipped			(RT=19.85)		150 - 500C: 0.05 600C - 800C: 0.04-0.05	
10V hipped			(RT=23.30)		150 - 500C: 0.07-0.05 600C - 800C: 0.04-0.05	
H13	RT-200F: 6.1x10-6/ degF RT-1200F: 7.3x10-6		(RT=23.70)		150 - 500C: 0.07-0.05 600C - 800C: 0.03-0.05	